Nova Scotia Research Foundation Corporation
Dartmouth, Nova Scotia

LIQUEFACTION OF NOVA SCOTIA COALS
PHASE III

STUDIES ON MEDIUM TO HIGH SEVERITY DONOR SOLVENT EXTRACTION

FINAL REPORT

by

NOVA SCOTIA RESEARCH FOUNDATION CORPORATION

Prepared by

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SUMMARY

The Third Phase of our experimental program was concentrated on high severity donor solvent liquefaction of coals from two operating mines in Cape Breton. Hydrogen donor solvent required for the bench-scale studies was prepared in a fixed-bed catalytic hydrotreater. Coal hydrogenation experiments were carried out in batch and continuous-flow systems.

Batch autoclave results for Lingan mine coal have shown that reaction temperature, pressure and residence time affect product composition. At high severity operating conditions, 450°C and 27.58 MPa, maf conversion was 89% while oil yield reached 38%. Increasing the residence time from 15 to 60 minutes at 450°C and 20.68 MPa had little effect on oil yield but it did significantly influence gas make. Additionally, it has been noted that Hub seam coal from the Prince mine is a more reactive feedstock at thermal liquefaction conditions than Lingan Harbour seam coal.

During Phase III the first version of our continuous coal liquefaction unit was commissioned. The duration of continuous donor solvent extraction experiments ranged from 3 to 8 hours. Studies were carried out varying dissolver temperature, coal feedstock and slurry feedrate. Based on the experimental results it has been concluded that ash and heavy reacting coal fragments experience prolonged hold-up in the tubular dissolver at low slurry feedrates.

During the next phase of work the continuous coal liquefaction unit will be extensively modified and upgraded. At first, the tubular dissolver will be replaced with a stirred tank reactor. Subsequently a weighed feed system, a hydrogen flow control system and an automated product let-down system will be added to the unit. Future operations will concentrate on processing Sydney area coals in both thermal and catalytic hydrogenation modes.

INTRODUCTION

Coal hydroliquefaction processes involve a number of mechanical and chemical steps which collectively alter the complex structure of coal *molecules*. When coal is heated to liquefaction temperatures, pyrolytic rupture of chemical bonds in the coal results in the formation of low-boiling materials and highly reactive free-radicals of heavy coal fragments. The free-radicals so produced can achieve stabilization by accepting hydrogen, in which case liquid product is formed, or by reacting with similar fragments to form a solid less reactive mass. In the presence of hydrogen donor molecules, hydrogen is abstracted from the donor molecule thereby stabilizing the free-radical.

Process studies relating to the direct liquefaction of Nova Scotia coals were initiated at the Nova Scotia Research Foundation Corporation in the latter part of 1977. The first two phases of experimental work were concentrated on producing coal liquids via low severity extraction-coking (1, 2, 3). In this approach hydrogen donor solvent is used under mild conditions (<2100 kPa, 400^{o}C) to extract the coal followed by delayed coking as a product separation step. Although the low pressure extraction has advantages related to plant hardware there is one major disadvantage, liquid yield is low. Experimental work showed that for one hour extraction conversions of Lingan coal to benzene solubles plus gases were typically 60%. However, following the coking step liquid yield was ca. 30 wt. %. A variety of methods were

investigated to enhance liquid yield from the process but results obtained were not encouraging. Hence it was decided to abandon the low severity approach in pursuit of higher yields of liquid products.

Phase III of our program began April 1, 1980. Experimental efforts were concentrated on medium to high severity donor solvent liquefaction. Donor solvent required for the test work was prepared using a fixed bed catalytic hydrotreater. Coal hydrogenation experiments were carried out in bench-scale batch and continuous-flow units.

Results of the batch autoclave coal hydrogenation experiments are presented in Task 4. The effects of temperature, pressure, time and solvent/coal ratio were investigated. Yields of oil, asphaltenes, preasphaltenes and gases were determined.

A description of the continuous coal liquefaction unit is given in Task 1. The apparatus was designed for processing 0.5-2.0 Kg per hour of coal oil slurry at pressures up to 28 MPa. Details pertaining to the commissioning and operation of this unit are presented herein.

TASK 1 - PRELIMINARY ACTIVITIES

1.1 Commissioning of Continuous Coal Liquefaction Unit

Actual construction of the basic continuous-flow coal hydrogenation unit was completed in March of 1980. Commissioning of the system began in April. A simplified flow diagram of the unit is presented in Figure 1. The second high pressure separator and the high pressure knock-out vessel shown in the drawing were added to the basic system in October (Task 6). The unit was designed for processing 0.5 - 2.0 Kg per hour of coal oil slurry at pressures up to 28 MPa.

In the system, coal and oil are mixed batchwise in a 16-liter feed vessel. Agitation is provided by an electric mixer as well as a rotary pump. The latter prevents settling of coal fines and provides suction pressure to the metering pump. Hydrogen is added and the mixture is fed through a fluidized sand bath preheater and then to an up-flow liquefaction column, 213 cm x 2.5 cm D (internal dimensions). The column is heated by a single-zone tube furnace. Downstream of the reaction zone products are passed to a hot separator. The overhead vapours and gases from the hot separator pass to a cold knock-out vessel. Light liquid products are condensed in the cold knock-out and a vent gas stream is provided through a pressure reducer regulator. The vent gas stream is monitored with a gas meter. The bottoms slurry from the hot separator is flashed at atmospheric pressure and collected.

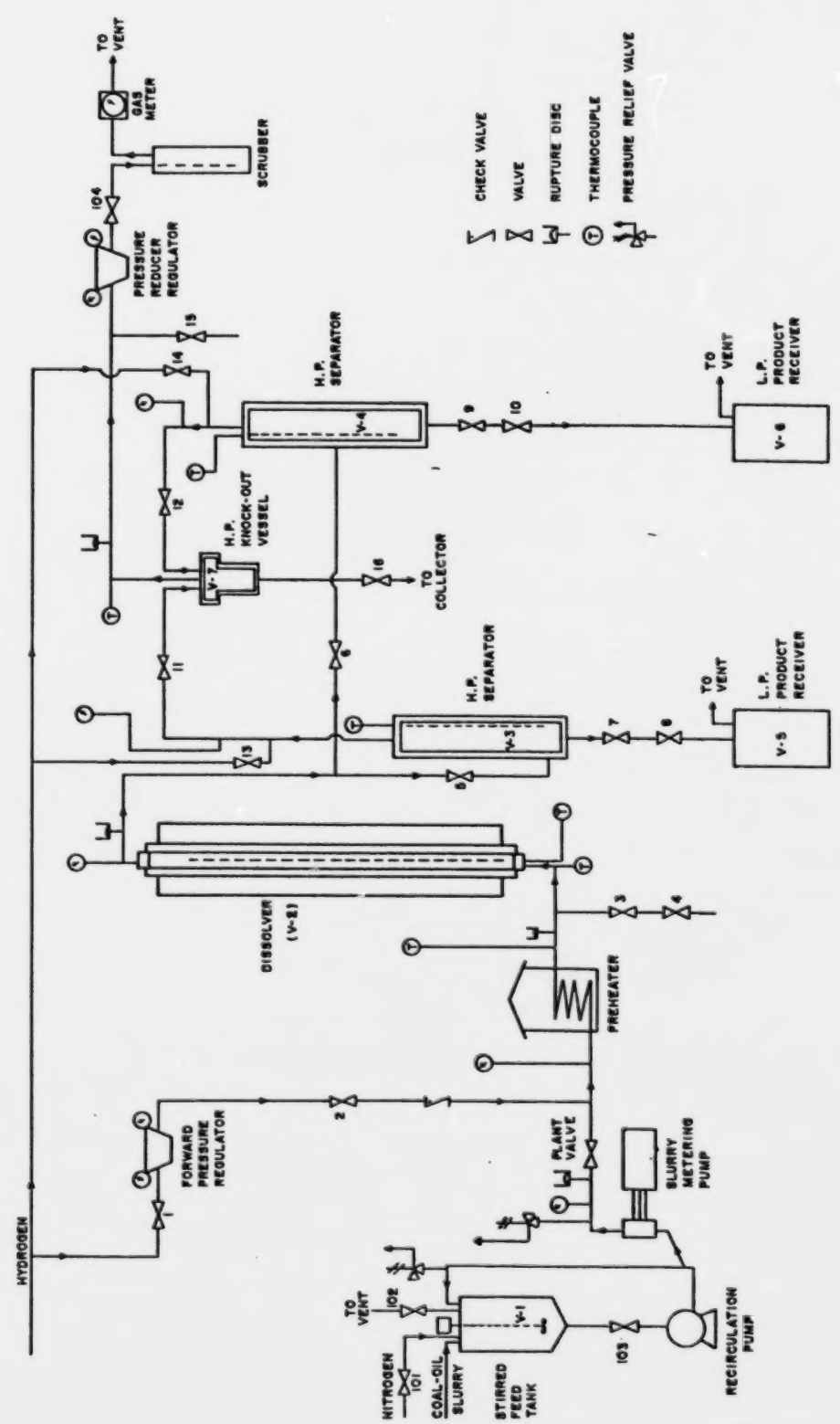


Figure 1 - FLOWSHEET FOR CONTINUOUS COAL LIQUEFACTION UNIT

Following initial pressure testing with argon and then hydrogen, trials were undertaken circulating hydrogenated anthracene oil through the unit. The operating temperature and pressure were increased stepwise over a series of runs to 450°C and 20.68 MPa. During these trials the discharge rate of the metering pump was found to be highly dependent on the degree to which the piston packing was tightened. Furthermore, on several occasions the metering pump failed to discharge and had to be reprimed. Loss of prime was attributed to gas bubbles in the pump suction line. The gas bubbles were generated by the vigorous stirring action in the feed tank. This difficulty was eliminated by reducing the speed of the motorized stirrer.

Severe pumping irregularities were experienced when coal was introduced to the system. These irregularities were attributed to solids fowling the operation of a check valve installed in the discharge line leading from the pump. This problem was eliminated by replacing the check valve with a manual shut-off valve, see plant valve in Figure 1. A positive shut-off valve is required at this location to permit priming of the metering pump if it becomes gas-locked.

During the commissioning trials the performance of the fluidized sand bath preheater was assessed in detail. It was established that the preheater be operated in the temperature range 250-300°C. Efforts were made to heat a 25 wt. % coal in oil slurry to higher temperatures by raising the temperature of the fluidized sand bath and by

increasing the length of the preheater coil. Unfortunately, in these trials the preheater coil plugged solid due to coke formation after only a few hours of operation.

1.2 Investigation of Stability and Viscosity of Coal Oil Mixtures

The purpose of this work was to gain knowledge of the stability and viscosity of coal oil mixtures. These properties are of particular interest when processing coal in oil because a highly unstable slurry undergoes rapid sedimentation of suspended particles causing excessive wear of pumps and valves, blockages of lines and related equipment and non-uniform flow.

A series of 24 sedimentation experiments were carried out varying coal concentration, particle size, vehicle oil and temperature. The tests were conducted with Lingan mine coal (Batch C) sized at -100 and -200 mesh. Raw anthracene oil, ca.5% H, and hydrogenated anthracene oil, ca. 9% H, were used as vehicles. Coal oil mixtures were prepared by adding each coal fraction to vehicle oil at concentrations of 20, 30 and 40 wt. %. The coal and oil were thoroughly agitated at 200 rpm for 20 minutes prior to initiating an experiment. It was originally planned to study the effect of temperature on stability at 25 and 90°C. However, the raw anthracene oil contained a substantial quantity of insoluble components at temperatures below 40°C. In consideration of this finding, sedimentation experiments with raw oil were carried out at 45 and 90°C.

The settling tests were conducted in a temperature controlled cabinet using standardized sedimentation columns. The column dimensions were 60 cm L x 4.4 cm ID. A sampling port with a 2 mm stopcock was located 24 cm from the bottom of the column. In this series of experiments the columns were filled to a height of 40 cm. Samples were withdrawn from the column ports at selected time intervals. After weighing, the samples were analyzed for solids content. This was accomplished by vacuum filtering the sample, washing the filter cake several times with benzene and then drying the solids to constant weight in a vacuum oven.

Viscosity measurements were made with a Brookfield RVT Viscometer at 20 rpm using spindle No. 1.

Sedimentation curves for mixtures of 20, 30 and 40% coal in oil are presented in Figures 2-11. The initial sedimentation rate varies inversely with the percentage of coal in the mixture. Thus a higher percentage of coal results in a lower sedimentation rate, that is, a more stable mixture. The main reason for a lower settling rate is the increased coal oil mixture viscosity at the higher coal concentrations. For example, the viscosity of a freshly prepared mixture of 20% coal (-200 mesh) in raw anthracene oil at 45°C was 67 cP whereas that of a 40% coal was 708 cP, see Figure 12. This increased mixture viscosity is due to a greater number of coal particle-particle interactions. These interactions are essentially inter-particle frictional forces which tend to slow down slippage of particles past one another.

Therefore, the settling rate of each coal particle is affected by the number and surface characteristics of the adjacent particles in the mixture.

Figures 2-5 show the effect of temperature on the settling behavior of -100 and -200 mesh coal in raw and hydrogenated anthracene oil. In general, coal particle sedimentation rates increase as the temperature of the mixture is raised. Increasing the temperature of the mixture also has the effect of decreasing its viscosity. For instance, the viscosity of 30% coal (-200 mesh) in hydrogenated anthracene oil at 25°C was 77 cP compared with 22 cP at 90°C. This decreased mixture viscosity at higher temperatures is responsible for the decreased mixture stability.

The effect of coal particle size on mixture stability is presented in Figures 6-9. The sedimentation curves show that stability increases with decreasing particle size. Viscosity/particle size relationships are shown in Figures 12-15. These figures illustrate that a reduction in coal particle size affects an increase in mixture viscosity.

Figures 10 and 11 show the effect of vehicle oil on mixture stability at 90°C. The sedimentation curves show that mixtures prepared with raw anthracene oil are more stable than those prepared with hydrogenated anthracene oil. However, it is interesting to note that at 90°C the viscosity of raw oil (10 cP) is similar to that of hydrogenated oil (7.8 cP). In consideration of this data it appears that chemical and/or molecular interactions between the solid and liquid phases also influence the stability of coal oil suspensions.

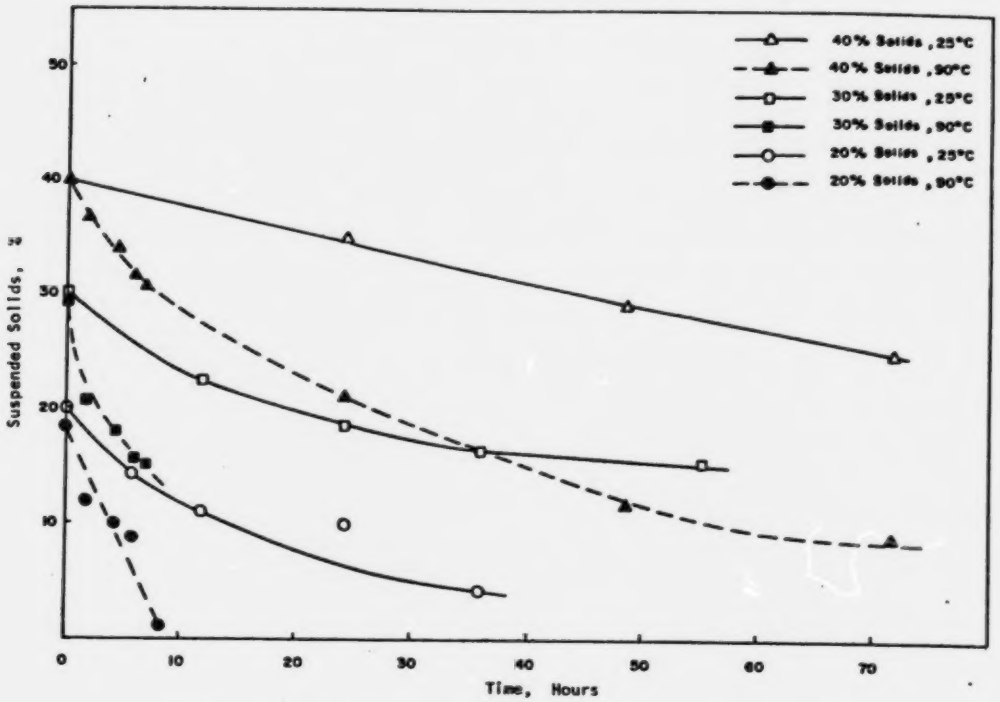


Figure 2 - EFFECT OF TEMPERATURE ON THE SETTLING BEHAVIOR OF -100 MESH LINGAN COAL IN HYDROGENATED ANTHRACENE OIL.

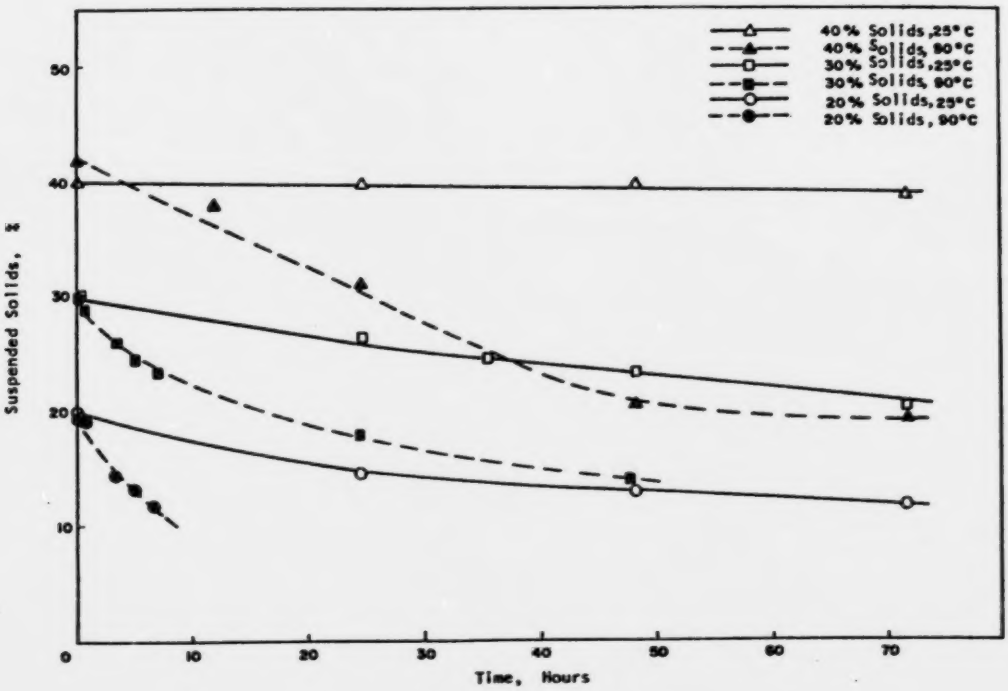


Figure 3 - EFFECT OF TEMPERATURE ON THE SETTLING BEHAVIOR OF -200 MESH LINGAN COAL IN HYDROGENATED ANTHRACENE OIL.

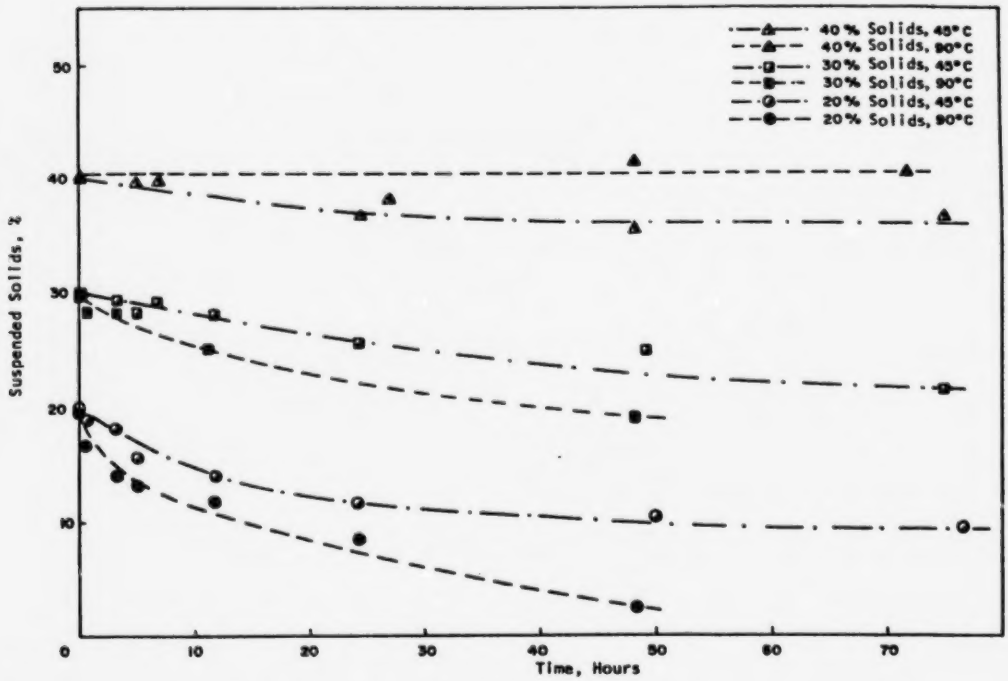


Figure 4 - EFFECT OF TEMPERATURE ON THE SETTLING BEHAVIOR OF -100 MESH LINGAN COAL IN RAW ANTHRACENE OIL.

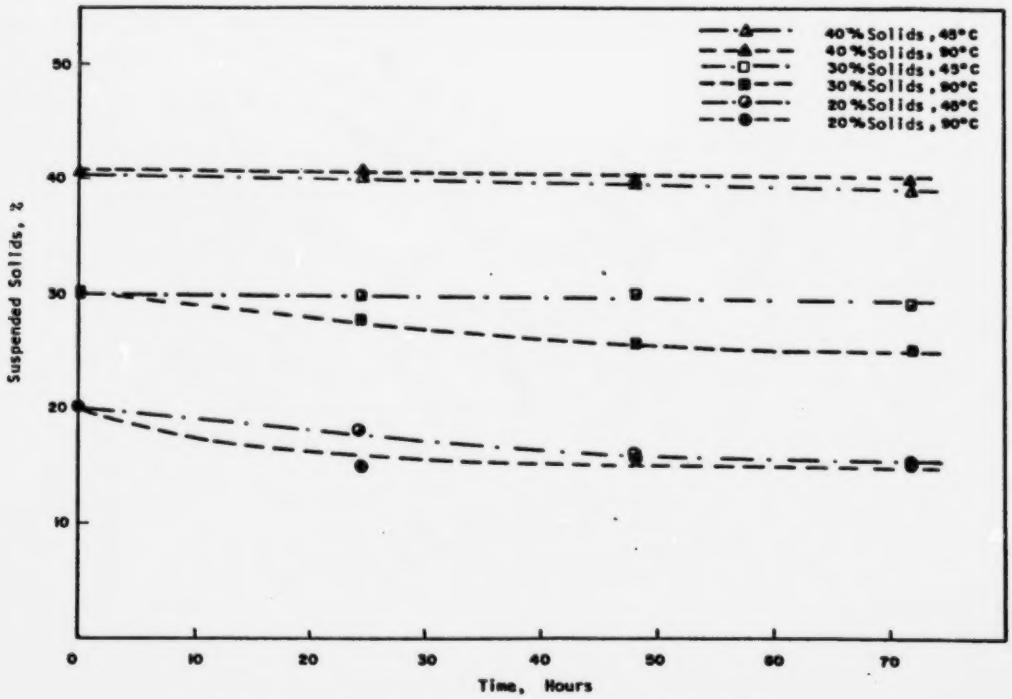


Figure 5 - EFFECT OF TEMPERATURE ON THE SETTLING BEHAVIOR OF -200 MESH LINGAN COAL IN RAW ANTHRACENE OIL.

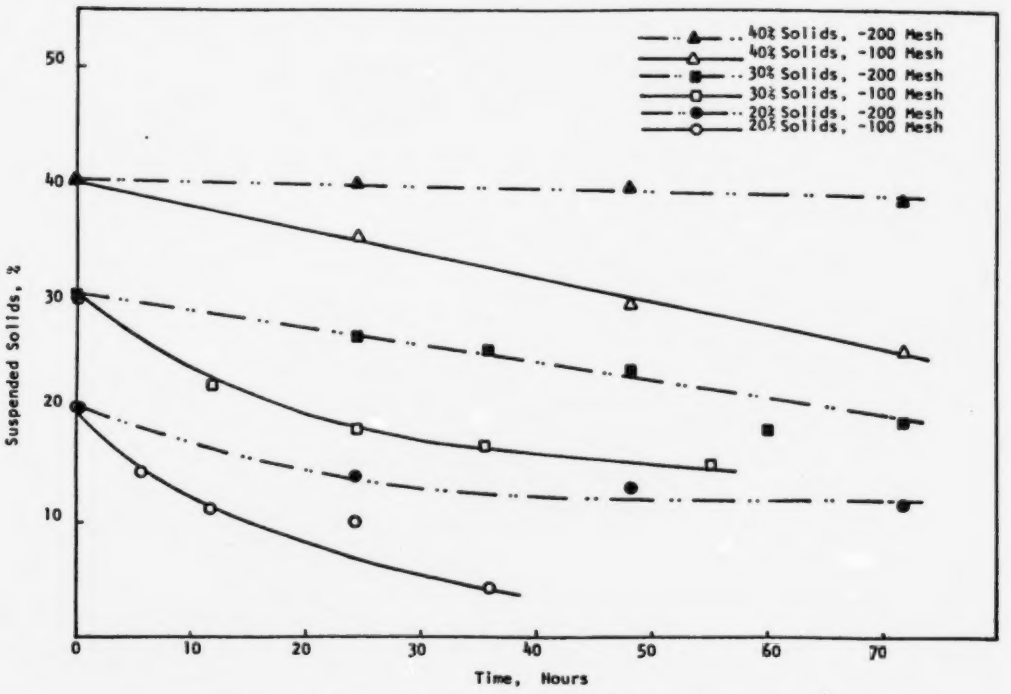


Figure 6 - EFFECT OF PARTICLE SIZE ON THE SETTLING BEHAVIOR OF LINGAN COAL IN HYDROGENATED ANTHRACENE OIL AT 25°C.

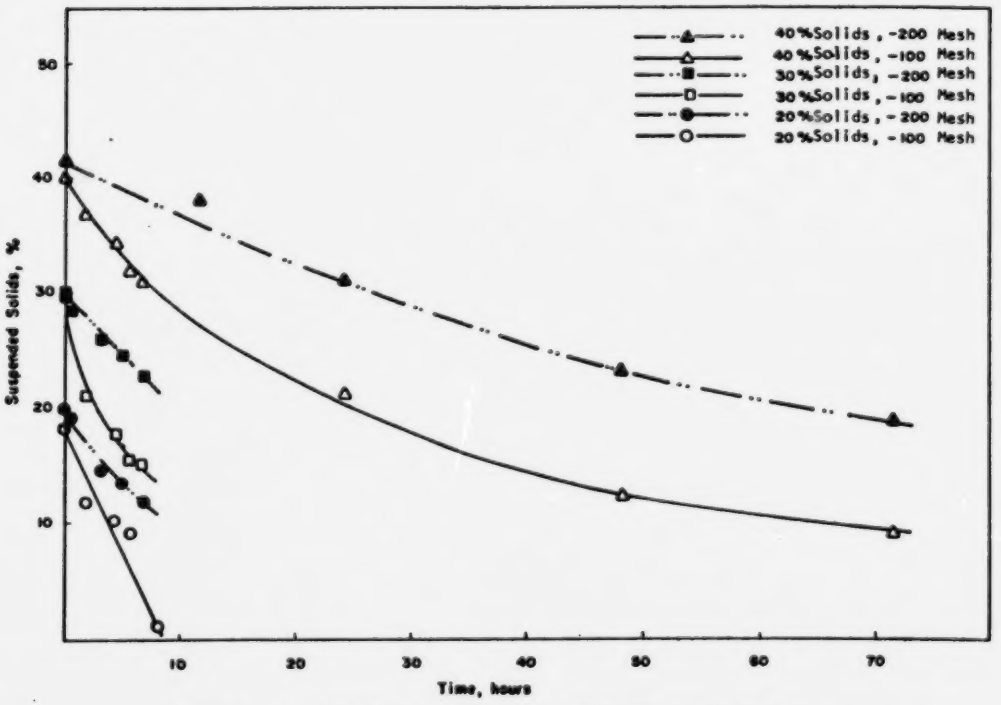


Figure 7 - EFFECT OF PARTICLE SIZE ON THE SETTLING BEHAVIOR OF LINGAN COAL IN HYDROGENATED ANTHRACENE OIL AT 90°C.

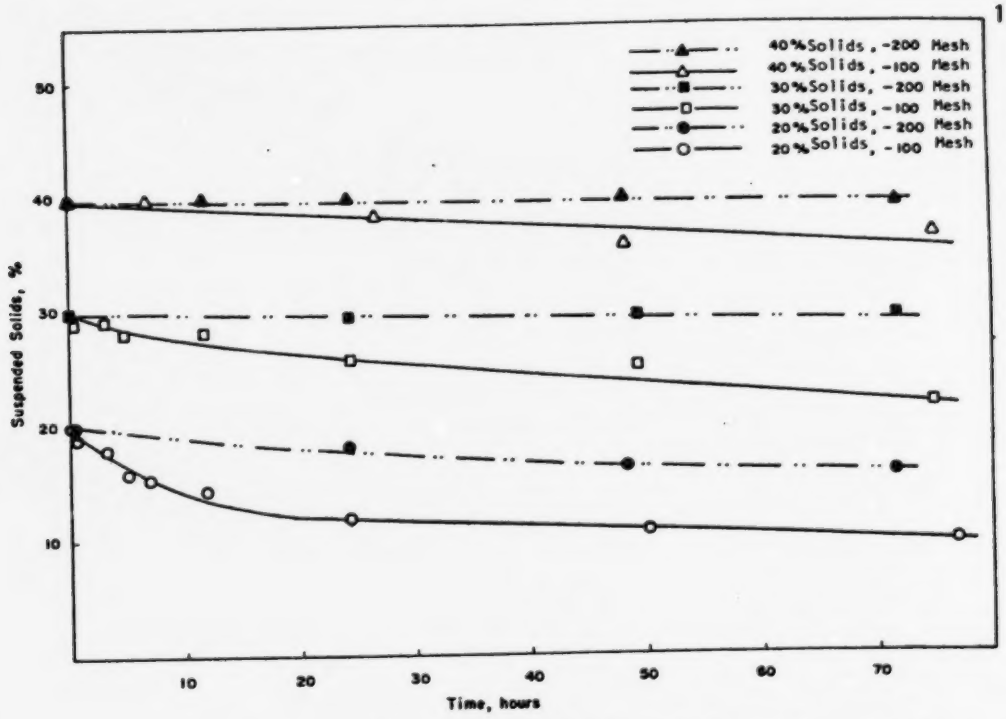


Figure 8 - EFFECT OF PARTICLE SIZE ON THE SETTLING BEHAVIOR OF LINGAN COAL IN RAW ANTHRACENE OIL AT 45°C.

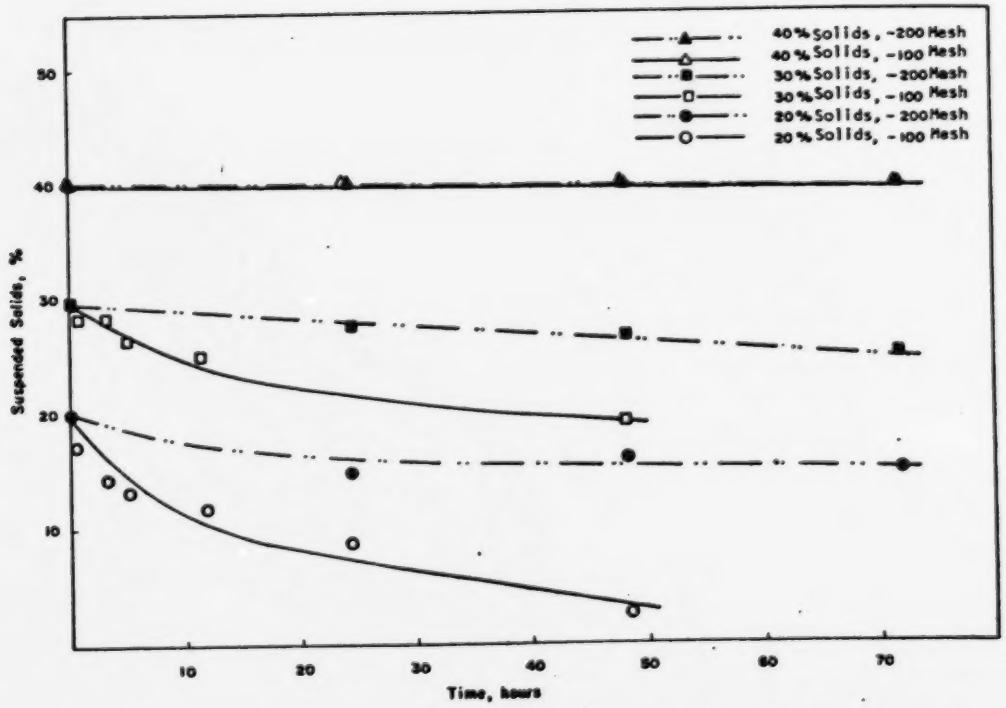


Figure 9 - EFFECT OF PARTICLE SIZE ON THE SETTLING BEHAVIOR OF LINGAN COAL IN RAW ANTHRACENE OIL AT 90°C.

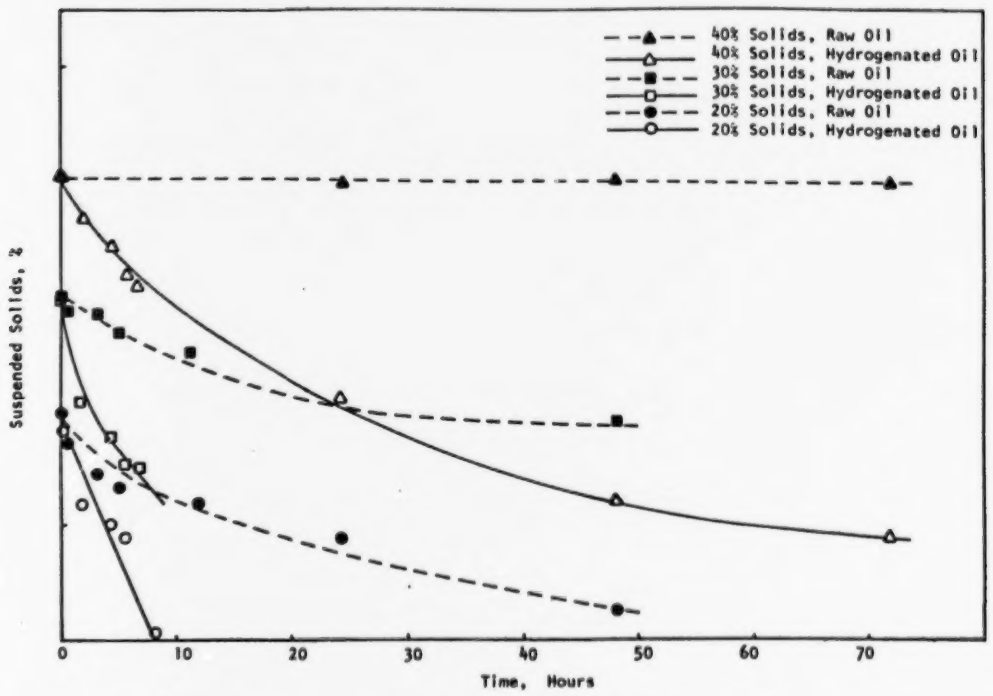


Figure 10 - SETTLING BEHAVIOR OF -100 MESH LINGAN COAL AS A FUNCTION OF VEHICLE OIL AT 90°C

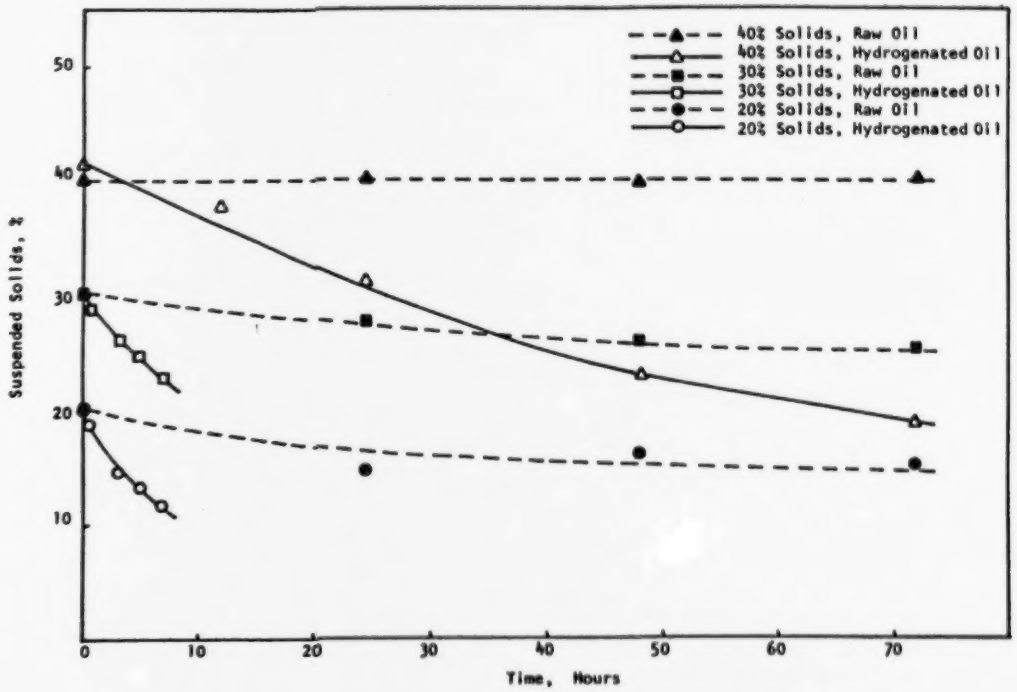


Figure 11 - SETTLING BEHAVIOR OF -200 MESH LINGAN COAL AS A FUNCTION OF VEHICLE OIL AT 90°C.

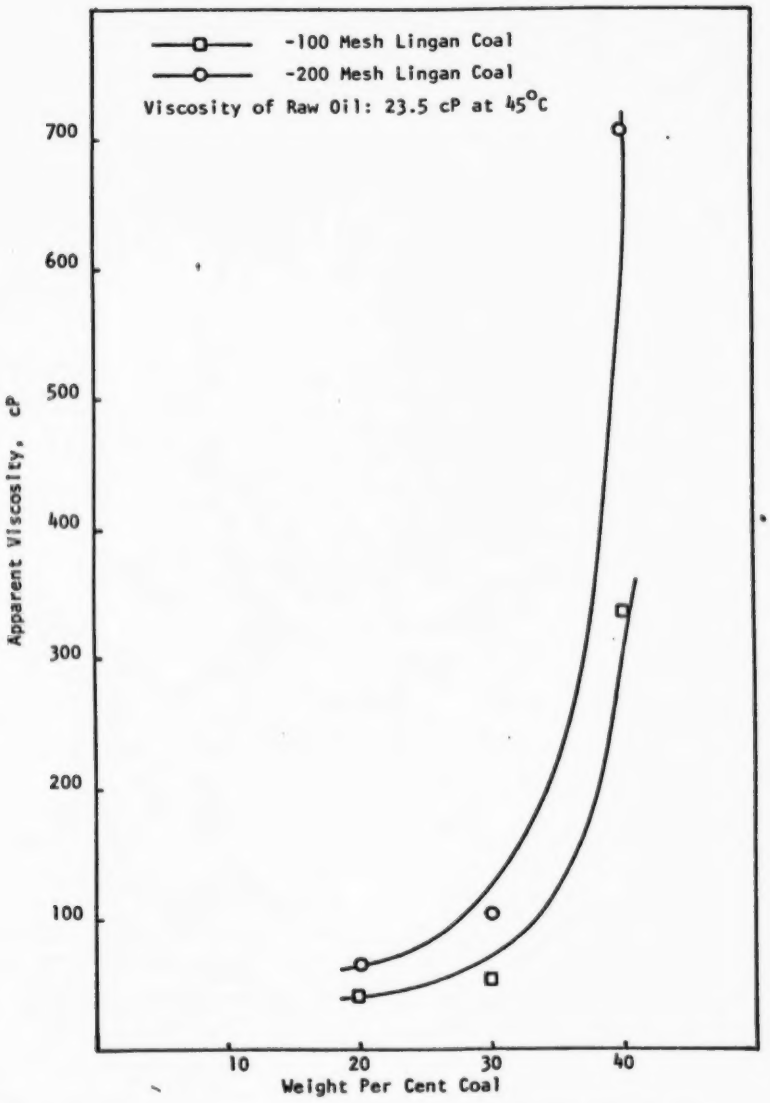


Figure 12 - INFLUENCE OF COAL CONCENTRATION AND PARTICLE SIZE ON APPARENT VISCOSITY OF COAL-RAW OIL MIXTURES AT 45°C

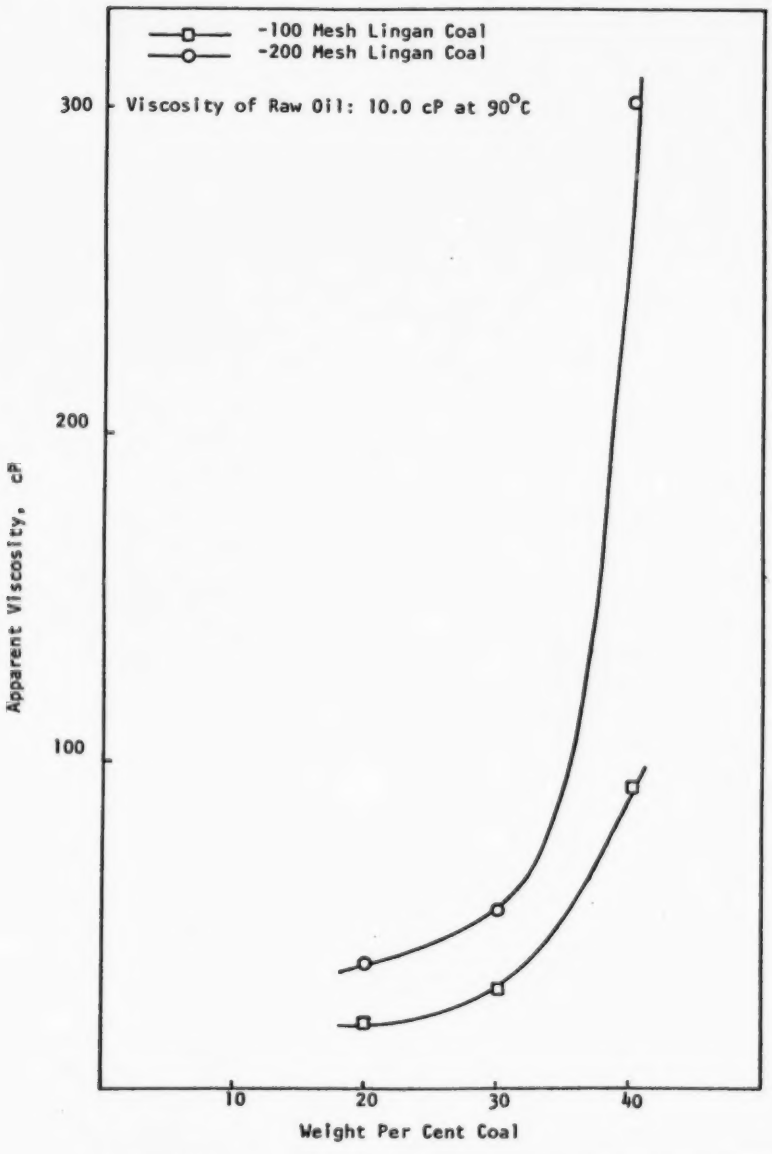


Figure 13 - INFLUENCE OF COAL CONCENTRATION AND PARTICLE SIZE ON APPARENT VISCOSITY OF COAL - RAW OIL MIXTURES AT 90°C

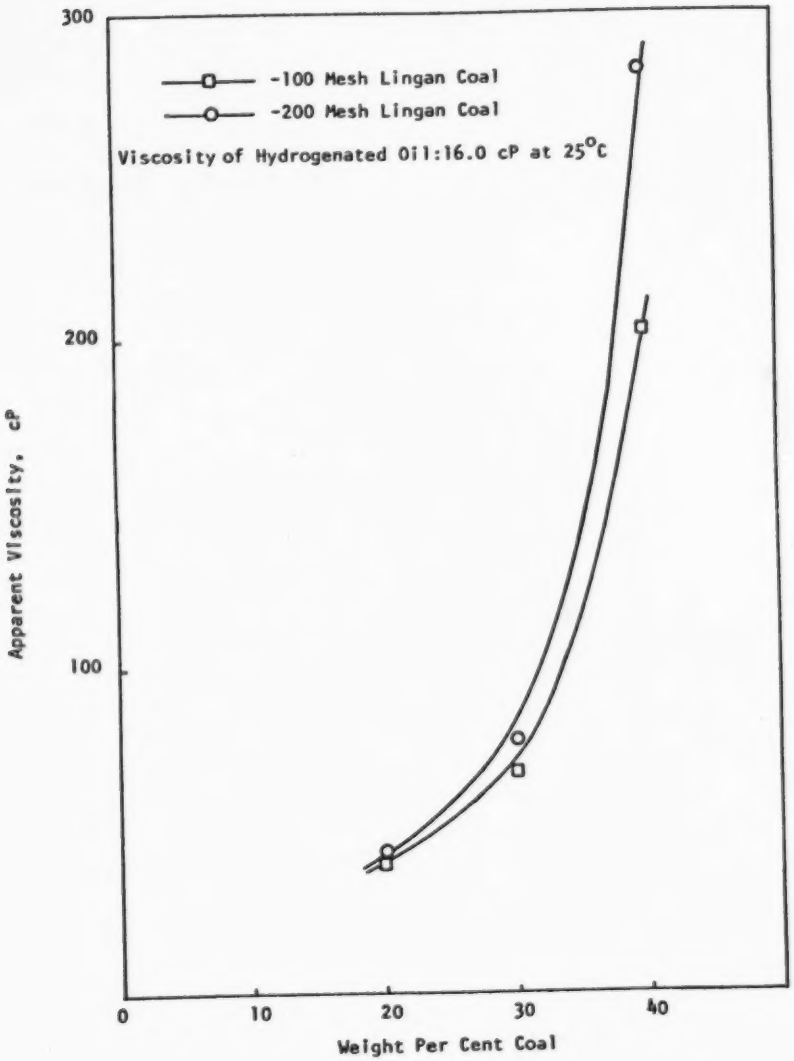


Figure 14 - INFLUENCE OF COAL CONCENTRATION AND PARTICLE SIZE ON APPARENT VISCOSITY OF COAL-HYDROGENATED OIL MIXTURES AT 25°C.

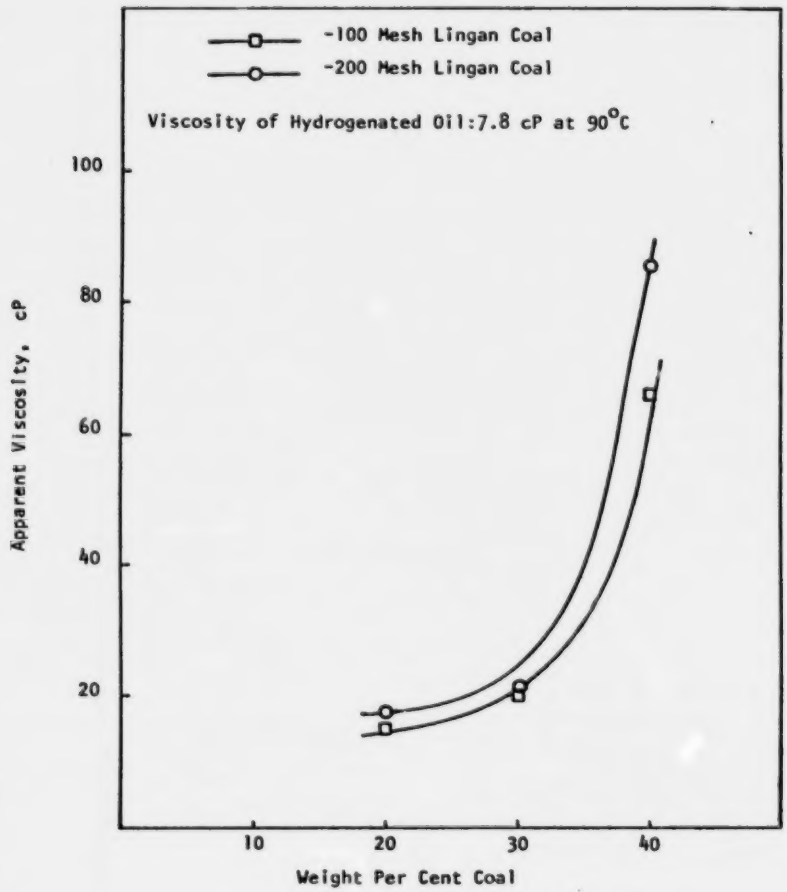


Figure 15 - INFLUENCE OF COAL CONCENTRATION AND PARTICLE SIZE ON APPARENT VISCOSITY OF COAL-HYDROGENATED OIL MIXTURES AT 90°C.

TASK 2 - PREPARATION AND ANALYSIS OF MATERIALS

2.1 Coal Feedstocks

Four coal feedstocks were used during this phase of the project. The coal samples were obtained from two operating mines in Cape Breton - Lingan and Prince. Preparation involved riffling, grinding and screening. After screening the individual coals were thoroughly mixed and stored in plastic-lined drums.

Proximate and ultimate analysis of the coals used are given in Table 1. Proximate analysis and forms of sulphur were determined according to standard methods. Total sulphur was measured using a Leco sulphur analyzer. Carbon, hydrogen and nitrogen were determined using a Perkin-Elmer elemental analyzer.

TABLE 1

ANALYSIS OF COALS TESTED

COAL DESCRIPTION:	LINGAN BATCH B	LINGAN BATCH C	LINGAN BATCH D	PRINCE BATCH B
Moisture, Wt. %	0.52	0.60	0.43	1.80
PROXIMATE (Dry Basis) Wt. %				
Ash	9.80	7.45	5.21	17.07
Volatile Matter	32.37	33.75	33.22	32.86
ULTIMATE (Dry Basis) Wt. %				
Carbon	75.79	77.18	78.86	60.91
Hydrogen	5.05	5.20	5.29	4.19
Nitrogen	1.73	1.53	2.13	1.85
Sulphur	1.84	1.81	2.38	6.68
Ash	9.80	7.45	5.21	17.07
Oxygen (by difference)	5.79	6.83	6.13	9.30
SULPHUR FORMS (Dry Basis) Wt. %				
Sulphate	0.06	< 0.01	0.05	0.48
Pyritic	1.39	1.40	1.95	5.69
Organic (by difference)	0.39	0.41	0.38	0.51
Total	1.84	1.81	2.38	6.68

2.2 Anthracene Oil

This material is a coal-tar distillate which contains ca. 5.5 wt. % hydrogen.

In March of 1980 a supply of raw anthracene oil was received from Domtar Chemicals Limited (Hamilton, Ontario). Difficulties were encountered with this batch of raw oil which have not been experienced with batches received in 1978 and 1979. On the basis of our earlier experience, the raw oil contained abnormally high proportions of low boiling (below 230°C) and high boiling (above 440°C) components. A typical yield on fractionation was as follows: 25 wt. % recovered below 230°C, 42 wt % between 230 and 440°C and 30 wt. % coke. Attempts to hydrogenate the 230-440°C fraction in our fixed-bed hydrotreater had to be abandoned due to extensive plugging of transfer lines between the feed vessel and the catalytic reactor.

Following a discussion with the manager of Domtar's Hamilton plant, a supply of raw aromatic oil suitable for our particular application was procured. Hydrogenated solvent used in the coal hydrogenation experiments was prepared from the second batch of oil received in May of 1980.

The characteristics of the second batch of oil are presented in Table 2. Carbon and hydrogen were determined using a Perkin-Elmer elemental analyzer. A standard Kjeldahl method was used for the determination of nitrogen. Total sulphur was measured by X-ray

fluorescence. The per cent of total hydrogen that occurs in different structural environments were determined by proton nuclear magnetic resonance (H-NMR). Figure 16 is a typical H-NMR spectrum of raw anthracene oil.

In this study H-NMR spectra were obtained using a Varian CFT-20 NMR spectrometer operating in the Fourier transform mode at 80 MHz and a Nicolet 360 NB NMR spectrometer operating in the Fourier transform mode at 360 MHz. In both cases tetramethylsilane was used as internal reference and peak areas were integrated electronically using software routines supplied by the manufacturers. Proton assignments were as follows: Aromatic hydrogens (H_{ar}) are found in the region 9-6 ppm. Benzylic hydrogens, including methyls, occur at 3.5 to 2 ppm (H_{α}). Hydrogens, except methyls, on carbons one or more positions removed from aromatic rings, occur at 2 - 1.1 ppm (H_{β}). Aliphatic methyls are found at 1.1 - 0.4 ppm (H_{γ}). As almost no long aliphatic side chains are believed to be present, the H_{β} protons are predominantly in rings. These assignments are consistent with similar analysis reported in the literature (4, 5).

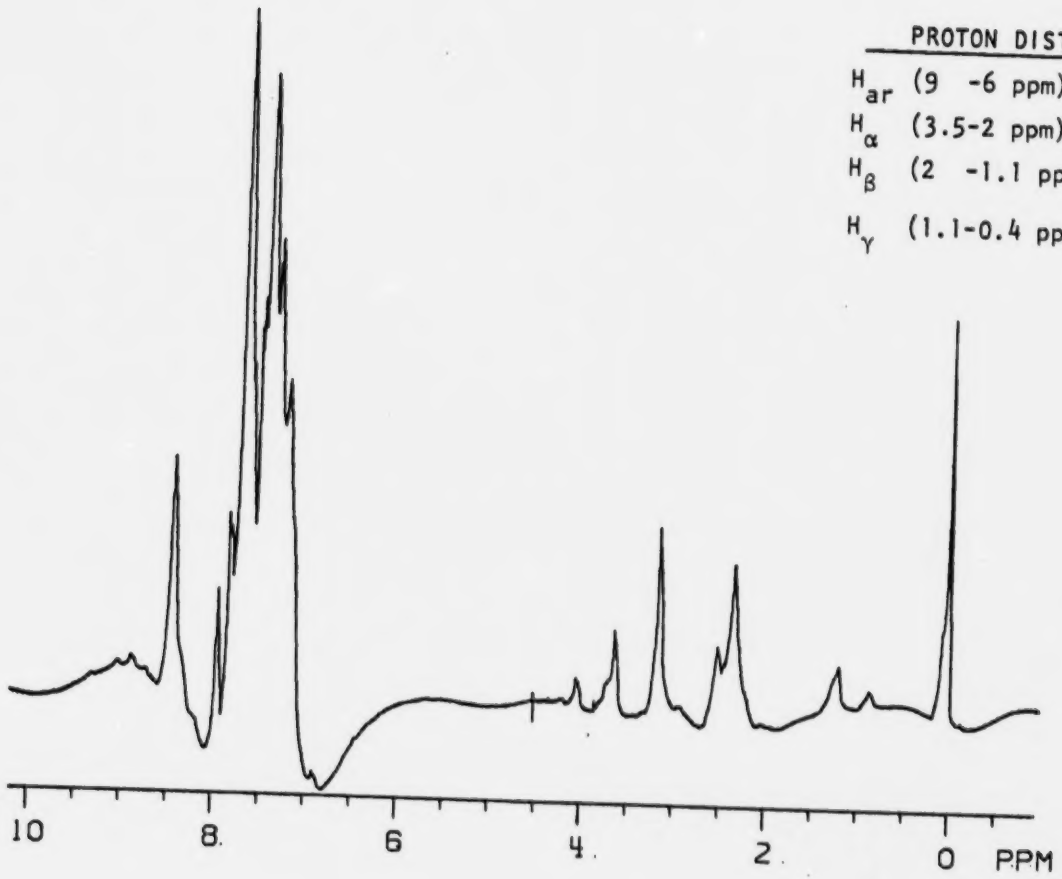
TABLE 2

ANALYSIS OF RAW ANTHRACENE OIL

Specific Gravity, 60/60°F	1.154
Carbon, Wt. %	91.71
Hydrogen, Wt. %	5.39
Nitrogen, Wt. %	0.87
Sulphur, Wt. %	0.78
Proton Distribution, %	
H_{ar} (9-6 ppm)	82.69
H_{α} (3.5-2 ppm)	14.13
H_{β} (2-1.1 ppm)	1.41
H_{γ} (1.1-0.4 ppm)	1.77
Distillation Data, °C (ASTM D-1160)	
IBP	238
10%	315
20%	337
30%	350
40%	360
50%	369
60%	381
70%	393
80%	413
90%	447
95%	470
EP	494

Figure 16 - H-NMR SPECTRA OF RAW ANTHRACENE OIL

PROTON DISTRIBUTION, %	
H_{ar} (9 -6 ppm)	82.69
H_{α} (3.5-2 ppm)	14.13
H_{β} (2 -1.1 ppm)	1.41
H_{γ} (1.1-0.4 ppm)	1.77



TASK 3 - PRODUCTION OF HYDROGEN DONOR SOLVENT

This task was an essential part of Phase III. Hydrogen donor solvent was prepared from raw anthracene oil using a fixed-bed catalytic hydrotreater. The bench-scale unit has been described in an earlier report (3). On the basis of our past experience, the quality of donor solvent is quite sensitive to the hydrotreating conditions employed in its preparation. Thus low severity extractive coking experiments were carried out to evaluate and monitor the donor capacity of solvents produced.

In our earlier work hydrogenated anthracene oil was fractionated to yield donor solvent with a boiling point range of about 230-440°C. Although the fractionation step can be simply achieved by batch distillation, this step adds considerable time to the overall procedure. Thus our initial efforts were aimed at preparing donor solvent which would not require fractionation.

Table 3 summarizes the results of preliminary anthracene oil hydrogenation trials. Experiments were carried out varying type of catalyst, temperature, liquid space velocity and hydrogen treatrate. Two commercial hydrotreating catalysts, Nalco NM502 and Nalcomo 479, were used. The chemical and physical properties of these Ni/Mo and Co/Mo catalysts are given in Table 4. The hydrogen content of solvents prepared ranged from 8.2 - 9.1%.

TABLE 3

RESULTS OF PRELIMINARY ANTHRACENE OIL HYDROGENATION TRIALS

RUN NO.	CATALYST	REACTOR TEMPERATURE (°C)	REACTOR PRESSURE (MPa)	HYDROGEN FLOW (L/Hr)	LHSV	HYDROGEN CONTENT (Wt. %)	
						PRODUCT SOLVENT	230 - 440°C FRACTION OF PRODUCT SOLVENT
1080	Nalco NM502	390	17.24	240	0.60	8.34	- -
1081	Nalco NM502	390	17.24	240	0.40	8.72	- -
1082	Nalco NM502	390	17.24	240	0.40	8.48	- -
1083	Nalco NM502	390	17.24	240	0.40	8.73	- -
1084	Nalco NM502	390	17.24	240	0.40	8.89	- -
1085	Nalco NM502	390	17.24	240	0.40	8.63	- -
1086	Nalco NM502	390	17.24	240	0.24	9.11	- -
1089	Nalcomo 479	390	17.24	300	0.20	8.20	8.17
1090	Nalcomo 479	410	17.24	300	0.20	8.82	- -
1094	Nalcomo 479	390	17.24	420	0.20	- -	8.85

TABLE 4

CHEMICAL AND PHYSICAL PROPERTIES OF HYDROTREATING CATALYSTS

	NALCO NM502	NALCOMO 479
MoO_3, Wt. %	14.0	19.0
NiO, Wt. %	4.0	
CoO, Wt. %		4.4
Size, Cm	0.16	0.16
Surface Area, M^2/g	240	180
Pore Volume, cc/g	0.53	0.47
Density, g/cc	0.73-0.74	0.82-0.84
Crush Strength, Kg	6.4 -7.3	6.4 -7.3

Nalco NM 502 and Nalcomo 479 are manufactured by the Nalco Chemical Company.

Extractive coking yield data for several of the solvents are presented in Table 5. An examination of Run Nos. 201-206 illustrates that the 230-440°C fraction of the hydrogenated solvents produced higher coal conversions and liquid yields than the full boiling range solvents. In view of these results it was decided to continue the earlier practice of fractionating the product solvent.

At this stage the routine preparation of hydrogen donor solvent began using the operating conditions of Run No. 1094. As the catalyst sulphided and aged the operating conditions were adjusted. Donor solvent containing ca. 9% H was prepared at the rate of 1.8 liters per 7 hour man-day. The hydrogenated oil so prepared was mixed to obtain 20-liter batches. Twelve 20-liter batches were prepared during this phase of the project. Analysis of the individual batches are given in Table 6. Extractive coking yield data for Batch Nos. 1, 2, 3, 4 and 10 are shown in Table 5. A proton NMR spectrum of 9.1% H solvent is presented in Figure 17.

TABLE 5

EXTRACTIVE COKING YIELD DATA FOR HYDROGENATED SOLVENTS PREPARED FROM ANTHRACENE OIL

RUN NO.	DESCRIPTION OF SOLVENT TESTED	SOLVENT HYDROGEN CONTENT (Wt. %)	GAS MAKE (g)	SOLIDS OUT (g)	CONVERSION OF maf COAL (Wt.%)	LIQUID YIELD, Wt. % OF maf COAL
201	Mixture of liquid products from Run Nos. 1084 and 1085	8.76	9.3	52.6	33	19
202	Liquid product from Run No. 1086	9.11	8.6	55.7	28	15
203	Liquid product from Run No. 1089	8.20	12.7	50.7	35	17
204	Liquid product from Run No. 1090	8.82	11.9	57.5	25	8
205	230-440°C fraction of liquid product from Run No. 1089	8.17	9.7	49.6	37	23
206	230-440°C fraction of liquid product from Run No. 1094	8.85	9.0	48.6	38	25
208	20-liter batch No. 1	8.96	9.5	47.9	39	26
210	20-liter batch No. 2	8.99	9.4	48.8	38	24
211	20-liter batch No. 3	8.99	10.4	47.7	40	25
212	20-liter batch No. 4	9.10	8.4	48.7	38	26
214	20-liter batch No. 10	8.99	8.8	48.8	38	24

Coal Description: Lingan Batch C (-100 mesh)
Coal Charged: 75.0g Solvent/Coal Ratio - 3:1
Extraction Conditions: 400°C for 1 hour.
Coking Conditions: 450-475°C for 1.5 hours.

TABLE 6

ANALYSIS OF 20-LITER BATCHES OF 9% H SOLVENT

BATCH NO.	1	2	3	4	5	6
Specific Gravity, 60/60°F	1.028	1.026	1.025	1.035	1.033	1.038
Carbon, Wt. %	88.95	88.94	89.91	90.32	90.56	90.14
Hydrogen, Wt. %	8.96	8.99	8.99	9.10	8.93	8.84
Nitrogen, Wt. %	0.06	0.06	0.08	0.11	0.12	0.13
Sulphur, Wt. %	0.004	0.003	0.007	0.002	0.006	0.010
Proton Distribution, %						
H_{ar} (9 -6 ppm)		25.37				28.94
H_{α} (3.5-2 ppm)		20.90				23.36
H_{β} (2-1.1 ppm)		42.54				35.52
H_{γ} (1.1-0.4 ppm)		11.19				12.18
Distillation Data, °C (ASTM D-86)						
IBP	192	199	197	194	163	197
5%	257	257	257	267	257	267
10%	276	273	274	280	271	280
20%	289	290	289	295	289	294
30%	299	299	299	306	301	306
40%	308	309	309	316	313	316
50%	317	316	317	324	321	325
60%	327	326	325	334	331	334
70%	337	337	336	347	342	347
80%	349	351	347	363	357	364
90%	367	371	367	390	382	390
95%	389	389	385	418	406	420
EP	493	494	463	>500	461	>500

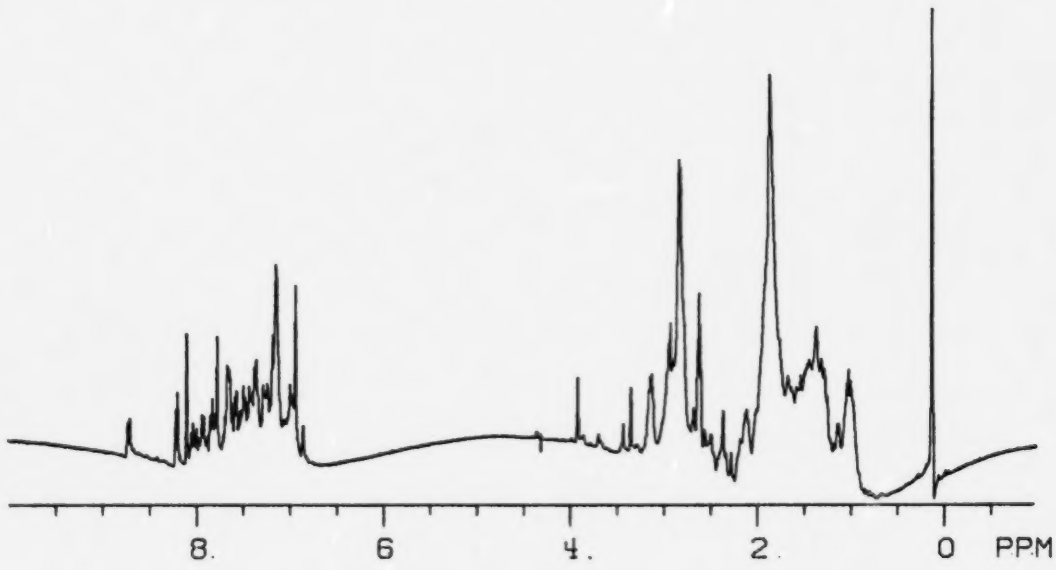
TABLE 6 (Continued)

ANALYSIS OF 20-LITER BATCHES OF 9% H SOLVENT

BATCH NO.	7	8	9	10	11	12
Specific Gravity, 60/60°F	1.034	1.035	1.033	1.035	1.036	1.036
Carbon, Wt. %	90.25	90.28	90.75	90.55	90.42	90.70
Hydrogen, Wt. %	9.03	9.07	9.01	8.99	8.73	8.94
Nitrogen, Wt. %	0.11	0.09	0.10	0.11	0.12	0.11
Sulphur, Wt. %	0.005	0.012	0.003	0.004	0.004	0.010
Proton Distribution, %						
H_{ar} (9-6 ppm)				27.95		
H^{α} (3.5 - 2 ppm)				22.79		
H^{β} (2-1.1 ppm)				40.44		
H_{γ} (1.1 - 0.4 ppm)				8.82		
Distillation Data, °C (ASTM D-86)						
IBP	186	190	182	203	200	220
5%	262	264	262	260	262	264
10%	275	279	277	278	276	276
20%	291	292	291	293	291	292
30%	303	304	306	304	304	304
40%	313	314	314	314	315	314
50%	323	323	323	323	324	323
60%	332	331	332	332	334	333
70%	343	345	344	345	348	346
80%	360	360	360	360	354	361
90%	382	388	384	385	390	388
95%	408	416	407	410	426	412
EP	457	> 500	448	> 500	450	470

Figure 17 - H-NMR SPECTRA OF HYDROGENATED ANTHRACENE OIL WITH 9.1 WT. % HYDROGEN

PROTON DISTRIBUTION, %		
H_{ar}	(9 -6 ppm)	26.95
H_{α}	(3.5-2 ppm)	24.11
H_{β}	(2 -1.1 ppm)	38.30
H_{γ}	(1.1-0.4 ppm)	10.64



TASK 4 - BATCH AUTOCLAVE EXPERIMENTS

The principal objective of Task 4 was to develop conversion data for Nova Scotia coal under donor solvent liquefaction conditions in a batch autoclave system.

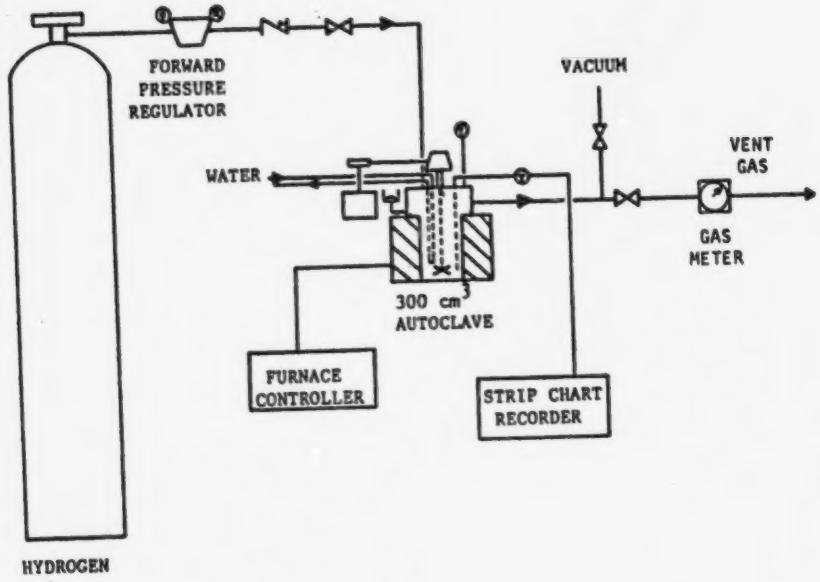
4.1 Procedures

A schematic diagram of the high pressure batch system is shown in Figure 18. The reaction vessel is an Autoclave Engineers 300 cm^3 magnedrive packless autoclave. The maximum allowable working pressure of the vessel is 35.16 MPa at 450°C. An internal cooling coil permits rapid cooling of the reaction products.

Each experiment was initiated by charging coal (-200 mesh) and hydrogenated anthracene oil (9.0% H) to the autoclave. The vessel was then sealed, evacuated and charged with hydrogen at room temperature to give the desired pressure at reaction temperature. Reaction temperatures ranged from 400 to 450°C. The total time required for heat-up was about 30-35 minutes. The reaction was carried out for selected time periods ranging from 15 to 60 minutes with the stirrer set at 600 rpm. Pressure was monitored throughout the test period with a dial gauge. Temperature was measured using a chromel-alumel thermocouple and a Philips recorder.

At the end of the reaction period the contents of the autoclave were rapidly cooled to room temperature. The vessel was depressurized by venting the gases through a wet test meter. The vent gas stream was sampled and its density was determined. At this stage the autoclave was opened and the product slurry was recovered with a modified pipette utilizing gentle suction.

Figure 18 - SCHEMATIC DIAGRAM OF HIGH PRESSURE AUTOCLAVE SYSTEM



In these experiments the following definitions for the product fractions isolated by solvent extraction were used:

Oil	-	pentane solubles
Asphaltenes	-	benzene solubles, pentane insolubles
Preasphaltenes	-	THF solubles, benzene insolubles.

A direct extraction technique was used to determine these fractions. The product slurry was thoroughly mixed and then split into three equal portions. The individual portions were separately extracted with pentane, benzene and tetrahydrofuran (THF). The duration of the Soxhlet extractions was 24 hours. Following this period the extration thimble and its contents were dried in a vacuum oven at 85°C. After weighing, the dried solids were ashed at 750°C. Conversions were calculated from an ash balance as shown in Appendix A.

Gas yield calculations were based on the mass of vented gases and the mass of hydrogen charged.

4.2 Results and Discussion

Table 7 summarizes the results from donor solvent liquefaction experiments carried out with Cape Breton coals in the high pressure batch system. Figures 19-22 are presented to illustrate the variation of coal conversion with reaction temperature, pressure, residence time and solvent to coal ratio.

The effect of reaction temperature on the conversion of Lingan coal is shown in Figure 19. The tests were conducted at 20.68 MPa for 60 minutes. Increasing the temperature from 400 to 450°C had no

effect on conversion to THF solubles plus gases. However, oil yield increased from 15 to 30% and preasphaltenes decreased from 25 to 9% as the temperature was raised from 400 to 450°C.

A series of five runs were made varying hydrogen pressure. The tests were conducted for 60 minutes at 450°C. Figure 20 illustrates that pressure, over the range 6.89 to 27.58 MPa, has little effect on conversion to THF solubles plus gases but it does influence oil yield significantly. The highest oil yield, 38%, was produced at 27.58 MPa.

Data presented in Figure 21 indicates that Lingan coal is converted to THF solubles plus gases rapidly at 450°C and 20.68 MPa in the presence of hydrogen donor solvent. Within 15 minutes 87% of the maf coal was converted to THF solubles plus gases. The gas yield was approximately 7%. Extending the residence time from 15 to 60 minutes had a marginal effect on oil yield, it increased from 26 to 30%. However, the gas make at 60 minutes was more than double the gas make at 15 minutes.

Conversion of coal as a function of solvent to coal ratio is presented in Figure 22. The tests were carried out for 60 minutes at 20.68 MPa and 450°C. Yields of oil, asphaltenes and preasphaltenes were quite similar at solvent to coal ratios of 3:1, 2.3:1 and 1.5:1.

TABLE 7

RESULTS OF BATCH AUTOCLAVE DONOR SOLVENT LIQUEFACTION EXPERIMENTS

RUN NO.	TEMP. (°C)	OPERATING PRESSURE (MPa)	TIME (min)	COAL[1] CHARGED (g)	SOLVENT/COAL RATIO (W/W)	THF SOLUBLES PLUS GASES, Wt.% OF maf COAL	BENZENE SOLUBLES PLUS GASES, Wt.% OF maf COAL	PENTANE SOLUBLES PLUS GASES, WT.% OF maf COAL	GASES, WT.% OF maf COAL
601	400	20.68	60	29.9	3:1	86	61	21	5.7
602	425	20.68	60	29.9	3:1	88	73	38	11.5
603	450	20.68	60	30.0	3:1	88	79	46	16.2
604	450	6.89	60	32.0	3:1	85	71	25	14.8
605	450	17.24	60	30.1	3:1	89	80	45	16.4
606	450	24.13	60	29.9	3:1	90	81	49	15.7
607	450	27.58	60	30.0	3:1	89	82	52	14.4
608	450	20.68	15	29.8	3:1	87	69	34	6.8
609	450	20.68	30	29.6	3:1	87	73	36	11.5
610	450	20.68	45	29.9	3:1	89	77	40	14.3
611	450	20.68	60	38.3	2.3:1	88	78	45	13.0
612	450	20.68	60	59.7	1.5:1	86	75	45	12.5
620	450	20.68	60	38.4	2.3:1	93	87	56	17.4

[1] Lingan Coal (Batch D) used in Run Nos. 601-612, Prince Coal (Batch B) used in Run No. 620.

Solvent Hydrogen Content: 9.0%.

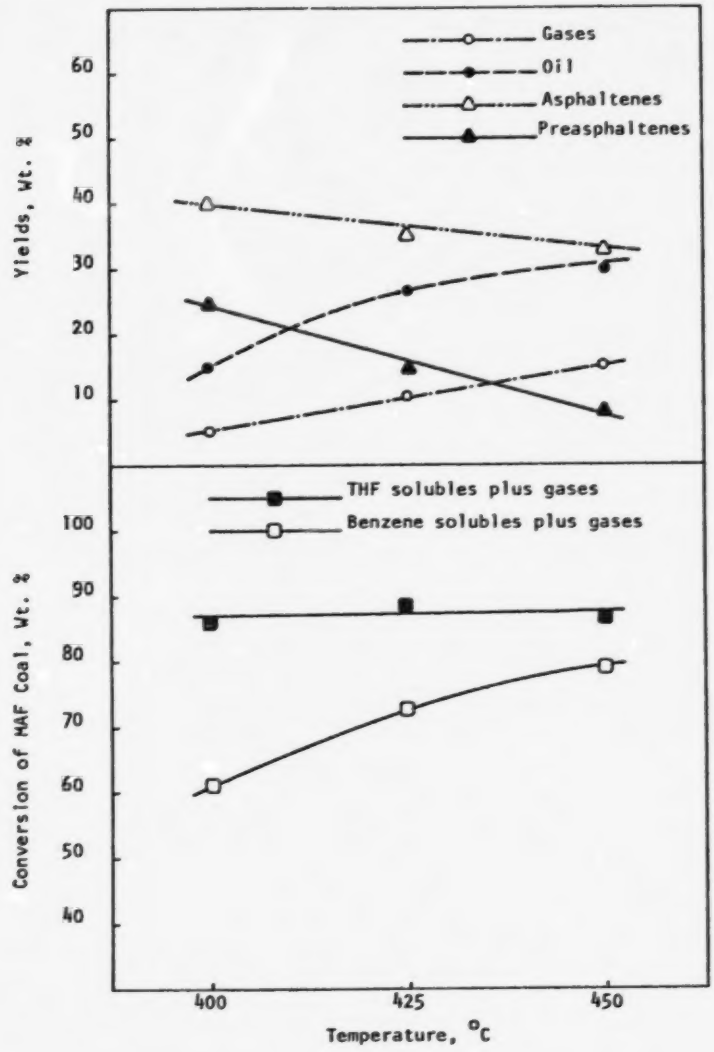


Figure 19 - EFFECT OF TEMPERATURE ON CONVERSION OF LINGAN COAL.

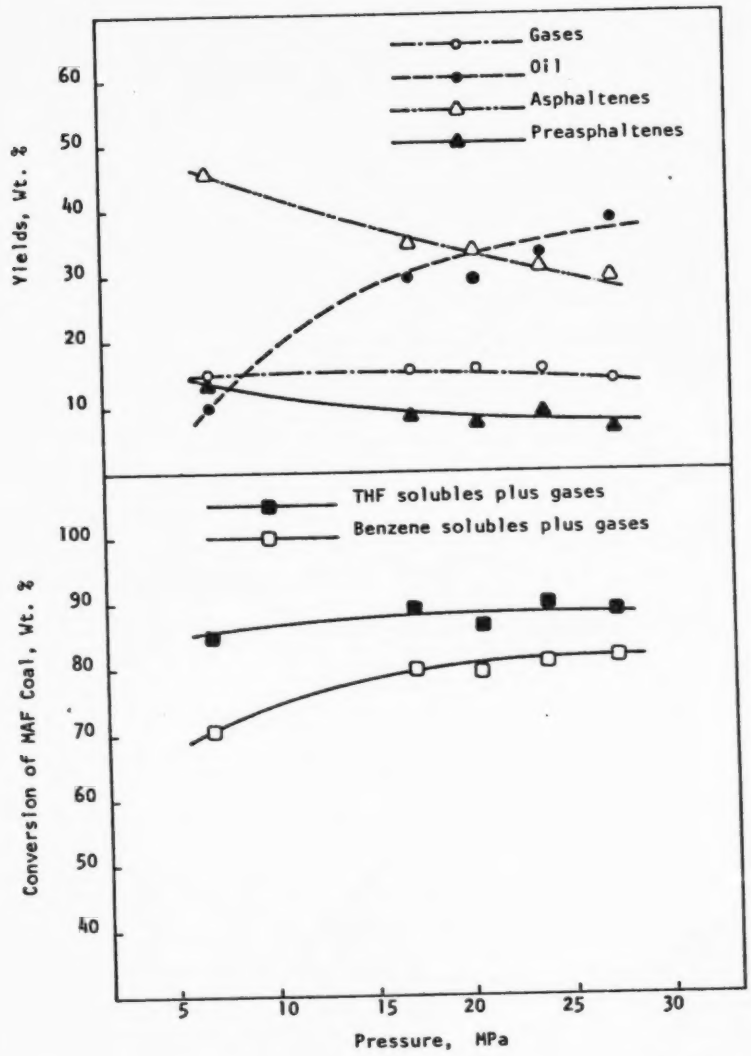


Figure 20 - CONVERSION OF LINGAN COAL AS A FUNCTION OF PRESSURE

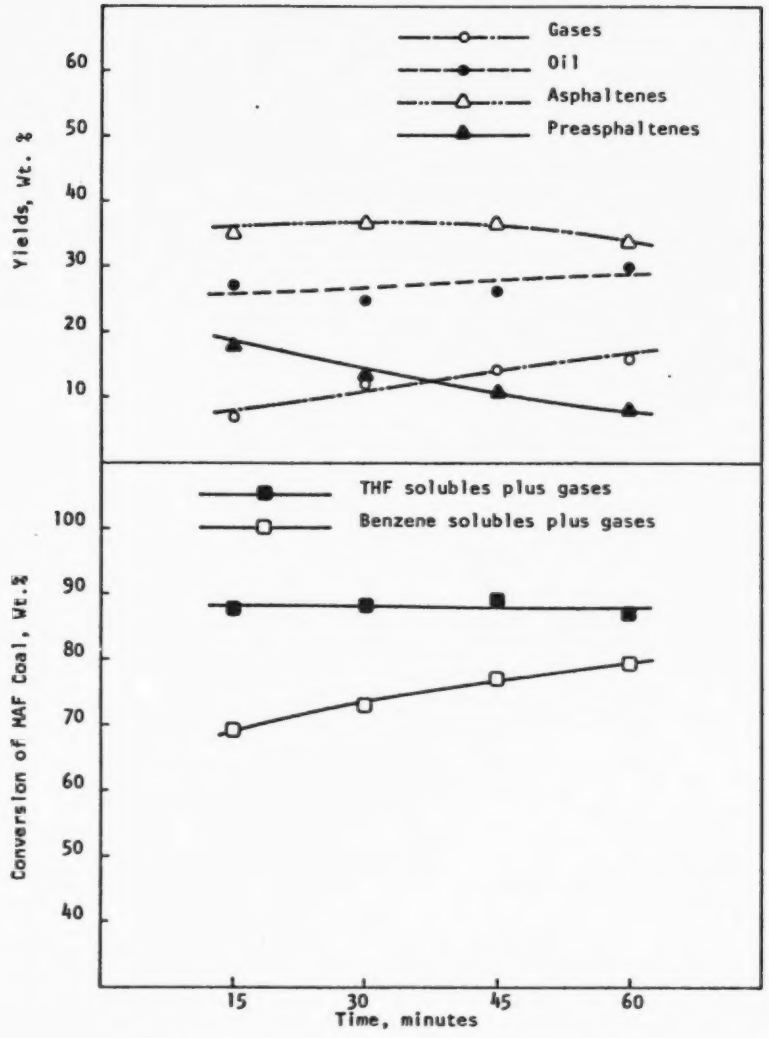


Figure 21 - EFFECT OF TIME ON CONVERSION OF LINGAN COAL

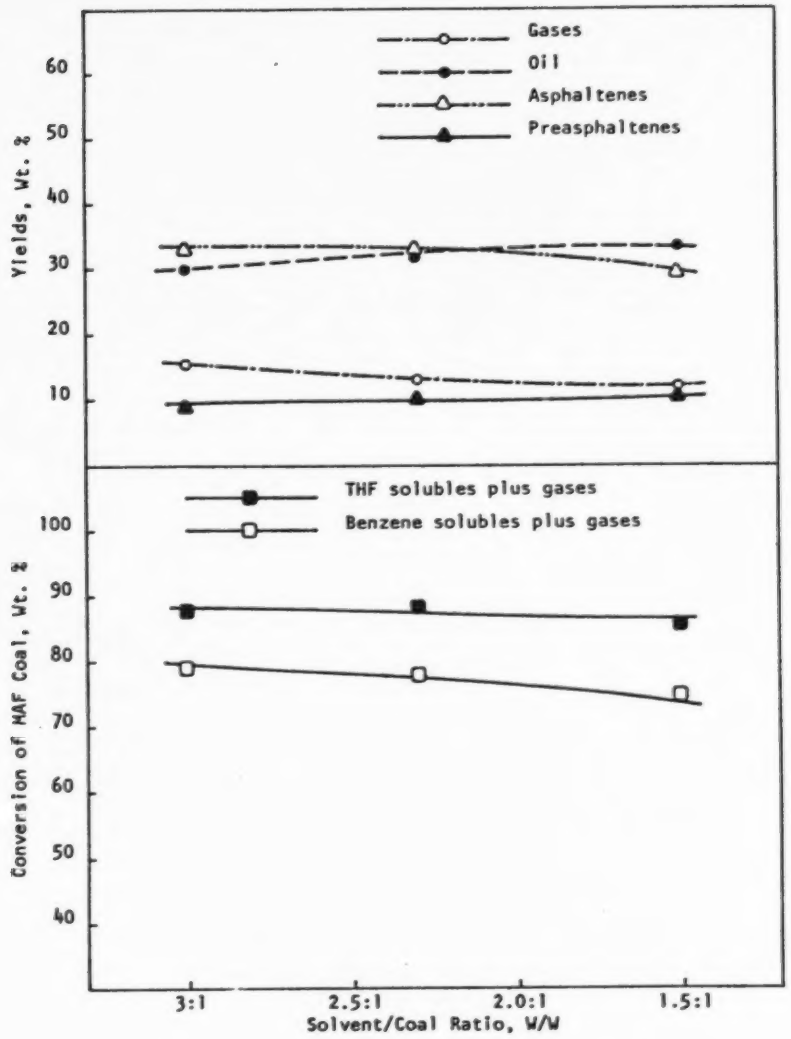


Figure 22 - VARIATION OF COAL CONVERSION WITH SOLVENT TO COAL RATIO.

A comparison of Run No. 620 with Run No. 611

(Table 7) shows that Prince mine coal is more reactive than Lingan coal at thermal liquefaction conditions. At the operating conditions specified, conversion of Prince coal to THF soluble plus gases was 93% compared with 88% for Lingan. In addition, oil yield was higher in the former case, 39 vs. 32%. The differences observed in conversion and oil yield are most likely related to the chemical and petrographic characteristics of the coals. Prince coal is typically higher in ash and sulfur content than Lingan. It is also a slightly lower rank coal than Lingan.

During this series of batch autoclave tests the direct extraction procedure worked as expected in most cases. However, it was observed that the slurry products from experiments at lower severity reaction conditions did not mix well with pentane. Consequently, oil yields for tests at 400°C and 6.89 MPa may be low. In any future experiments at similar operating conditions oil will be separated from the benzene soluble fraction using a large excess of pentane.

TASK 5 - OPERATION OF THE CONTINUOUS COAL LIQUEFACTION UNIT

The principal objective of this task was to develop yield data for Nova Scotia coals at donor solvent liquefaction conditions in the continuous bench-scale unit shown in Figure 1. A test program was designed to investigate the effects of operating conditions and coal properties on yield structures. The duration of continuous coal hydrogenation experiments ranged from 3 - 8 hours. However, an additional 5-6 hours was required for preliminary and post run operations.

Prior to installing the second high pressure separator, see Task 6, runs were of 3 hours duration. Initially experiments were carried out at 20.68 MPa varying the dissolver temperature from 400 to 450°C. The preheater coil used was 170 cm L x 6.4 mm ID. The preheater exit temperature was maintained at 275°C. Hydrogenated anthracene oil, 9% H, was used as the donor vehicle. The concentration of coal in the feed slurry was fixed at 25 wt. %. Slurry feedrate was ca. 1 Kg/hr and hydrogen treatrate was ca. 5 wt. % based on dry coal.

Figure 23 shows the effect of reaction temperature on conversion of Lingan 9.8% ash coal. In these experiments conversion, as the percentage of maf coal converted to gases and benzene soluble materials (oil and asphaltenes) was calculated from an ash balance. The figure shows that increasing the dissolver temperature from 400 to 450°C increased conversion from 48 to 65%. On closer examination of the experimental data, however, it was found that the absolute quantity of ash in the slurry product from the separator was low relative to the quantity

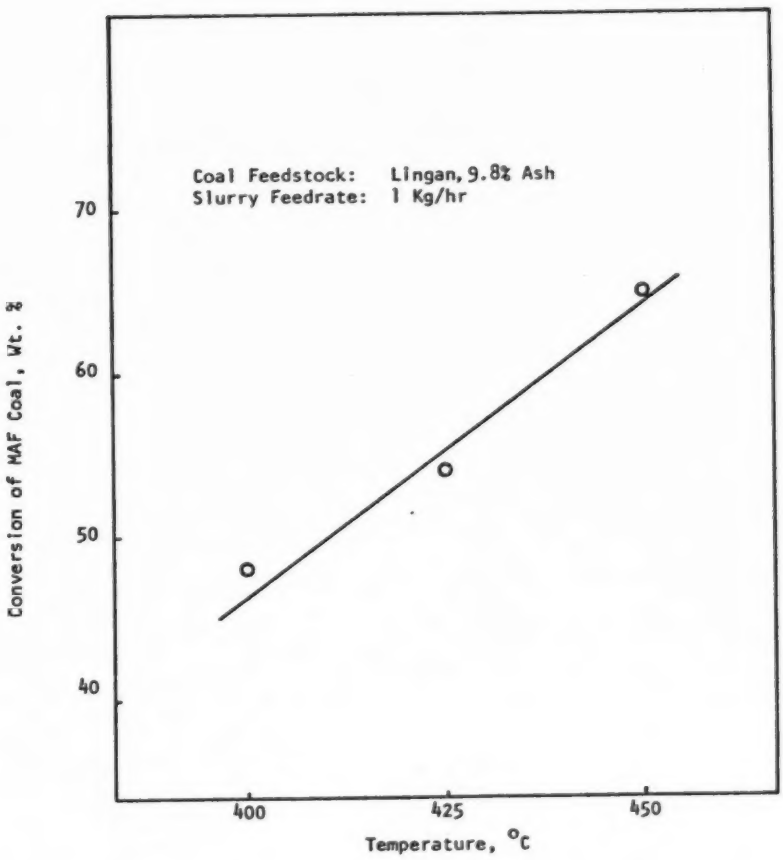


Figure 23 - EFFECT OF DISSOLVER TEMPERATURE ON CONVERSION OF LINGAN COAL.

of ash fed to the dissolver. As a consequence of this finding test work was initiated to investigate mass transport in the continuous-flow system.

The first step taken was to determine the amount of ash in the dissolver following an experiment. This was accomplished by hot discharging the partially reacted coal oil slurry from the dissolver into a 2-liter pressure vessel immediately after a run. The pressure vessel was installed between the preheater and the dissolver. Two manual shut-off valves were used to isolate this vessel from the system during a run. A short series of 3-hour experiments showed that ash accumulation occurs in the up-flow tubular reactor at low slurry feedrates. For example, twice the estimated amount of ash was found in the dissolver after a run at 450°C and a slurry feedrate of 1 Kg/hr.

Following the installation of the second separator experiments were carried out to study ash transport as a function of operating time. The first 6-hour run was completed without difficulty. However, problems related to the product withdrawal valves were encountered during the second run. Although the run was completed, the product let-down valves had deteriorated to the point where they had to be replaced after the run. These valves were made of 316 stainless steel. Following each product withdrawal operation extra force and a determined effort were required to achieve a gas tight shut-off when the valve was returned to its closed position.

On examination of the valves it was found that grooves were cut in the stems parallel to the slurry flow. The use of stellite

trim prolonged the service life of product let-down valves. Nevertheless, a second manual shut-off valve was installed downstream of the primary let-down valve as a safeguard against possible valve failure during a run.

Figures 24 and 25 illustrate the variation of ash transport with coal feedstock and slurry feedrate. In these experiments the reactor effluent was collected by alternating from one separator to the other. Each vessel was on-line for 45-60 minutes. Ash transport was determined for each period by calculating the quantity of ash in the product slurry as a percentage of the amount of ash fed to the reactor during that period. The calculated values were then plotted at the half-time point of the sampling period. Runs were carried out at 20.68 MPa and 450°C. The preheater was operated at 250-275°C. The concentration of coal in the feed slurry was fixed at 30 wt. %.

Figure 24 shows that when processing 9.8% ash coal at a slurry feedrate of 1 Kg/hr ash transport exceeds 95% after 5 hours on stream. However, in the case of 5.2% ash coal only 62% of the feed ash was passing through the reactor after 7.5 hours of operation. Data presented in Figure 25 indicates that ash hold-up in the dissolver is significantly affected by slurry feedrate when processing the low ash coal. For example, at a slurry feedrate of 1.6 Kg/hr ca. 75% of the feed ash was passing through the reactor after 5 hours compared with 47% at 0.5 Kg/hr.

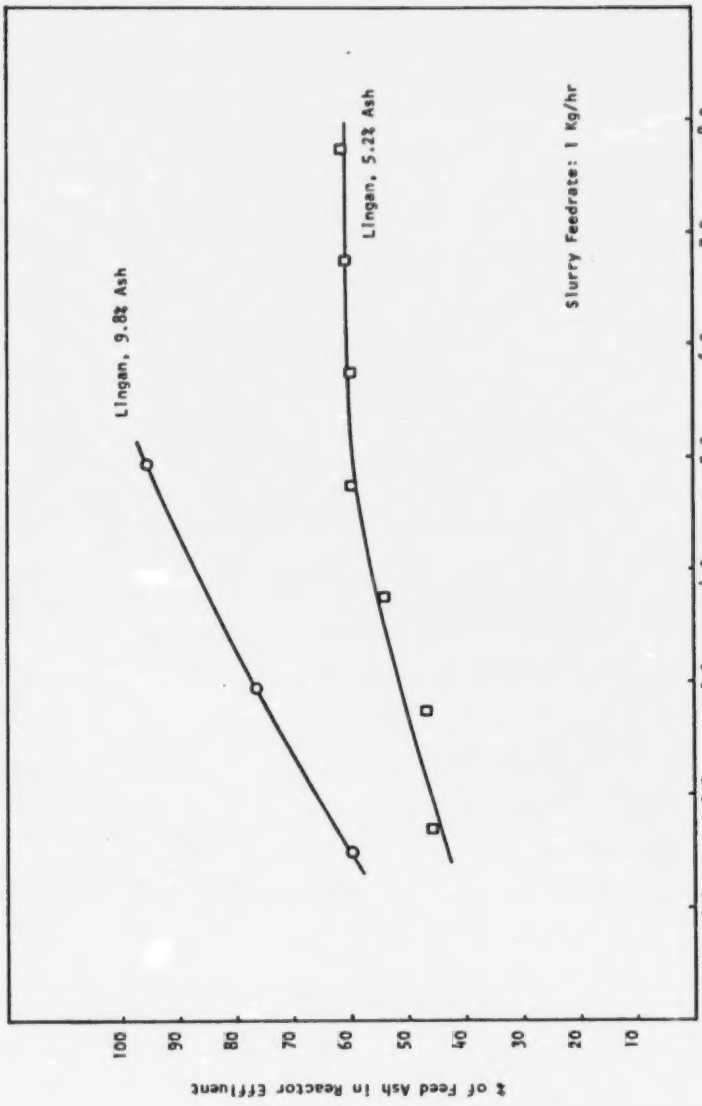


Figure 24 - VARIATION OF ASH TRANSPORT WITH COAL FEEDSTOCK

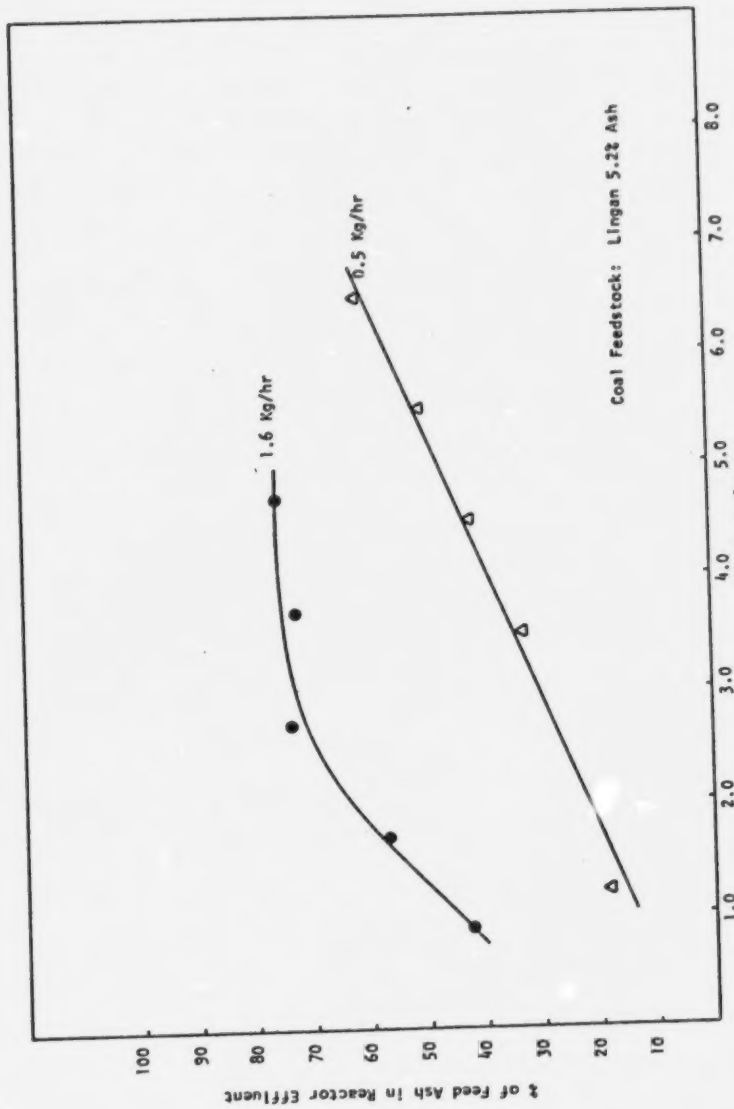


Figure 25 - VARIATION OF ASH TRANSPORT WITH SLURRY FEEDRATE

Our original intention was to conduct continuous experiments of 6-8 hours duration. However, these trials indicate that longer on-stream periods are required to achieve steady state operations. Furthermore, the test results suggest that heavy reacting coal fragments and unreacted coal associated with ash components experience prolonged hold-up in the reaction zone at certain operating conditions. Thus in order to control residence time and achieve steady state operations in runs of short duration it is necessary to modify the unit.

Two possible alternatives have been considered. Firstly, incorporate a recirculation loop into the existing tubular reactor system. Secondly, replace the tubular dissolver with a stirred tank reactor. In considering the severity of the process and the scale of the unit, the second approach is preferable because it is significantly less complex and offers a lower risk with regard to operability.

TASK 6 - PROCUREMENT AND INSTALLATION OF NEW EQUIPMENT

The purpose of Task 6 was to add new components to the basic continuous-flow coal hydrogenation unit. Activity centered around the installation of a second high pressure separator and a high pressure knock-out vessel.

Modifications to the rig were carried out during the fourth quarter of 1980. In addition to installing the two pressure vessels; appropriate tubing, valves and gauges were added to the system to permit switching from one separator to the other during a run. A flow diagram of the modified unit is shown in Figure 1. During a typical run the off-line separator is isolated in order to withdraw slurry product from the vessel without upsetting flows in the system. Before bringing the off-line vessel back on-stream it is charged to system pressure with hydrogen.

After the system was commissioned with hydrogenated anthracene oil at severe operating conditions, high temperature heating tapes were installed on the separators. These additions were made to facilitate the recovery of high viscosity slurry products. Presently the separators can be operated at temperatures up to 250^{o}C.

Details pertaining to the pressure vessels installed are given below:

Separator

A standard 2-liter vessel constructed of 316 stainless steel, purchased from Autoclave International Corporation, Burlington,

Ontario. The vessel has a design pressure rating of 62 MPa at 300°C. A standard AE closure is provided which is fitted with a thermowell and a gas outlet port. Additional ports are located at the side and the bottom of the vessel.

Knock-Out

A standard 1-liter vessel (bolted closure) constructed of 316 stainless steel, supplied by Autoclave International Corporation. The vessel has a design pressure rating of 38.5 MPa at 350°C. Three ports are located at the top of the vessel, a fourth at the bottom center.

CONCLUSIONS AND RECOMMENDATIONS

Lingan Harbour seam coal was evaluated in a batch autoclave system at medium to high severity donor solvent liquefaction conditions. Hydrogenated anthracene oil, 9% H content, was used as donor vehicle. As a result of these experiments it was found that:

(a) Temperature has a significant effect on product composition. As the temperature was raised from 400 to 450°C oil yield increased from 15 to 30% whereas preasphaltenes decreased from 25 to 9%.

(b) Pressure, over the range 6.89 to 27.58 MPa, has little effect on the conversion of coal to THF solubles plus gases. However, hydrogen pressure does influence oil yield substantially. The highest oil yield, 38%, was produced at 27.58 MPa.

(c) Residence time, over the range 15 to 60 minutes, has little influence on oil yield but it does affect gas make significantly. The gas make at 60 minutes was more than double the gas make at 15 minutes.

(d) Yields of oil, asphaltenes and preasphaltenes are quite similar for solvent to coal ratios of 3:1, 2.3:1 and 1.5:1.

Based on a limited number of experiments it has been noted that Hub seam coal from the Prince mine is a more thermally reactive feedstock than Lingan Harbour seam coal. At similar operating conditions conversion of Prince coal to THF solubles plus gases was 93% compared with 88% for Lingan. Furthermore, the Prince coal yielded 39% oil whereas the oil yield for Lingan was 32%. The differences observed in conversion and oil yield are most likely related to the chemical and petrographic characteristics of the coals.

In earlier phases of our experimental work efforts were concentrated on low severity (<2100 kPa, 400^{o}C) donor solvent liquefaction. The main purpose of increasing severity at the extraction stage was to enhance liquid yield. In consideration of the oil yields at 20.68 MPa, it is recommended that batch autoclave testing with catalysts be initiated. The use of catalysts offers potential benefits related to enhanced liquid yields and/or lower severity operating conditions. It is recommended that a series of potential hydroliquefaction catalysts be investigated to evaluate their hydrogenation activity as well as their denitrogenation and desulphurization activities.

During Phase III the continuous coal liquefaction unit was commissioned. This apparatus was designed for processing 0.5 - 2.0 Kg/hr of coal oil slurry at pressures up to 28 MPa. The duration of continuous donor solvent experiments ranged from 3 - 8 hours. Studies

were carried out with Lingan 5% and 10% ash coals. It was established that ash transport is dependent on the feed coal and the slurry feedrate. As a consequence of these findings it was concluded that ash and heavy reacting coal fragments experience prolonged hold-up in the dissolver at low slurry feedrates.

In order to control the residence time of coal particles in the reaction zone and achieve steady state operating conditions in runs of 6-8 hours, it is recommended that the up-flow tubular reactor be replaced with a stirred tank reactor. It is also recommended that a weighed feed system, a hydrogen flow control system and an automated product let-down system be added to the unit. The weighed feed system and the hydrogen flow control system will provide the degree of control required to generate reliable data. The automated product let-down system will facilitate operating the unit and permit accurate measurement of product gases.

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APPENDIX A

CONVERSION CALCULATIONS

APPENDIX A

CALCULATIONS FOR BATCH DONOR SOLVENT LIQUEFACTION EXPERIMENTS

CONVERSIONS, WT. %

Pentane solubles plus gases $= \frac{(A-a) \times 10^4}{A\ (100-a)}$

Where A = ash in pentane insoluble residue
and a - ash in dry coal

Benzene solubles plus gases $= \frac{(A-a) \times 10^4}{A\ (100-a)}$

Where A = ash in benzene insoluble residue
and a = ash in dry coal

THF solubles plus gases $= \frac{(A-a) \times 10^4}{A\ (100-a)}$

Where A = ash in THF insoluble residue
and a = ash in dry coal

Gases $= \frac{\text{Product gases}}{\text{maf coal charged}} \times 100$

DEFINITION OF PRODUCTS

Oil = Pentane solubles
Asphaltenes = Benzene solubles - pentane solubles
Preasphaltenes = THF Solubles - benzene solubles.

APPENDIX A

CALCULATIONS FOR EXTRACTIVE COKING TESTS

$$\text{Conversion} = \frac{(\text{Coal charged}) - (\text{Solids Out})}{\text{maf coal charged}} \times 100$$

$$\text{Liquid Yield} = \frac{(\text{Liquids Out}) - (\text{Solvent Charged})}{\text{maf coal charged}} \times 100$$

APPENDIX B

OPERATING PROCEDURE FOR CONTINUOUS COAL LIQUEFACTION UNIT

PREPARATION OF UNIT PRIOR TO ACTUAL OPERATION

This procedure is normally carried out the day before a run. At this time all valves are closed.

1. Prepare V-7 product withdrawal line for venting.
2. Open valves 5, 6, 11, 12 and 16 to depressurize the system.
3. Prepare V-7 product withdrawal line for vacuum. Turn on the vacuum pump.
4. After one hour close valve 16 and then shut off the vacuum pump.
5. Open valve 1.
6. Open valve 2 and pressurize the system to 13.78 MPa, close valve 2. Leave for two hours and observe the pressure.
7. Adjust the forward pressure regulator to the desired setting.

ACTUAL OPERATION

Start-Up

1. Set the desired operating temperature on the dissolver furnace controller. Activate the controller.
2. Energize the heating tapes on the separators and slurry transfer lines to desired voltage levels.
3. Charge V-1 with the coal oil slurry.
4. Prepare V-1 for vacuum. Open valve 102 and turn on the vacuum pump.
5. After 15 minutes close valve 102 and then turn off the vacuum pump.
6. Open valve 101 and purge the feed tank with nitrogen. Open valve 102 one quarter of a turn.
7. Open valve 103 and turn on the circulating pump.

8. Open valve 2 and pressurize the system to the desired operating pressure.

9. When the temperature of the dissolver is at the desired level, purge the high pressure metering pump with slurry until it is gas-free.

Run

10. Open the plant valve and activate the high pressure metering pump.

11. Immediately close valve 6.

12. After 10 minutes open valve 104 slightly, adjust the second stage of the pressure reducing regulator to 55 kPa and set the vent gas flowrate.

13. After a preselected operating period open valve 6 and close valve 5 to direct slurry product to V-4. Close valve 11.

14. Manually withdraw slurry product from V-3 by opening valve 8 and then slowly opening valve 7, close valve 7 and then 8. After 15 minutes repeat this procedure.

15. Add hydrogen to V-3 by opening valve 13 until the pressure is 207 kPa higher than system pressure. Close valve 13 and slowly open valve 11 to equalize system pressure.

16. After a preselected operating period direct slurry product from V-4 to V-3. To do this open valve 5 and close valve 6. Close valve 12.

17. Manually withdraw slurry product from V-4 by opening valve 10 and then slowly opening valve 9. Close valve 9 and then valve 10. After 15 minutes repeat this procedure.

18. Add hydrogen to V-4 by opening valve 14 until the pressure is 207 kPa greater than system pressure. Close valve 14 and slowly open valve 12 to equalize system pressure.

19. Repeat steps 13 through 18 as desired.

20. At end of run turn off the dissolver temperature controller and the separator heaters. Reduce voltage to heating tapes on slurry transfer lines.

21. Close valve 2.

22. Deactivate the metering pump.

23. Close the plant valve and valves 5, 11, and 104.

24. Transfer contents of V-2 into an evacuated pressure vessel through valves 3 and 4. Close both valves after one hour.

25. Repeat step 14.

26. Manually withdraw product from V-7 by slowly opening valve 16. Close valve 16.

27. Deactivate the circulating pump and drain slurry from the feed tank.

28. Open valve 11.

29. Open valve 2 and add hydrogen to the dissolver until its pressure is 689 kPa in excess of the downstream pressure and then close valve 2.

30. Open valve 5 and equalize the pressure throughout the system.

Flush Cycle and Shut-Down

31. Reactivate the dissolver temperature controller, the separator heaters and increase voltage to the heating tapes on slurry transfer lines to desired levels.

32. Close valve 103, and add flush oil to V-1, open valve 103 and turn on the circulating pump.

33. Purge the high pressure metering pump with oil until the liquid is gas-free. Open the plant valve and activate the metering pump.

34. Open valve 104.

35. When 1.5 Kg of oil has been pumped into V-3, open valve 6 and close valve 5 to transfer oil to V-4. Close valve 11.

36. Withdraw oil from V-3. Slowly equalize the pressure through valve 11.

37. After 1.5 Kg of oil has been pumped into V-4, open valve 5 and close valve 6 to direct oil to V-3. Close valve 12.

38. Withdraw oil from V-4. Slowly equalize the pressure through valve 12.

39. After 1.5 Kg of oil has been pumped into V-3, open valve 6 and close valve 5 to direct oil to V-4. Close valve 11.

40. Withdraw oil from V-3. Slowly equalize the pressure through valve 11.

41. After 1.5 Kg of oil has been pumped into V-4, deactivate the metering pump. Close the plant valve and valves 11, 12, 6 and 104.

42. Shut off all heating tapes, heaters and the dissolver temperature controller.

43. Transfer contents of V-2 into an evacuated pressure vessel through valves 3 and 4. After 15 minutes close valves 3 and 4.

44. Drain flush oil from V-1.

45. Open valves 11 and 12 to equalize the pressure in V-3, V-4 and V-7. Close valve 1.

POST-RUN OPERATIONS

1. Open valve 1.

2. Open valve 2 and pressurize the dissolver to 10.34 MPa and then close valve 2.

3. Transfer contents of V-2 into an evacuated pressure vessel through valves 3 and 4. After 15 minutes close valves 3 and 4.

4. Open valve 13 and pressurize V-3, V-4 and V-7 to 10.34 MPa and then close valve 13.

5. Close valves 11 and 12 and withdraw residual flush oil from V-3 and V-4. Close valve 1.

6. Evacuate the system by following steps 1 to 6 on page B-2.